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PLAN OF ACTION
for
CONFIRMATION STUDY ON
HAZARDOUS WASTE SITES
at
NAVAL EDUCATION AND TRAINING CENTER
NEWPORT, R.I.

November 4, 1983

Prepared for:

Engineering Facilities Division
Northern Division
Naval Facilities Engineering Command
Building 77-L, U.S. Naval Base
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Comm. No. 502-10

A/E Contract No. N62472-83-C-1154

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SECTION A - INTRODUCTION

1. SCOPE OF THE PLAN

This Plan of Action covers the initial phase of work (verification stage) of the Confirmation Study on Hazardous Waste Sites for the Naval Education and Training Center (NETC), Newport, Rhode Island. The work described herein will be carried out under A/E Contract No. N62472-83-C-1154 by Loureiro Engineering Associates of Avon, CT with laboratory analyses and other support being provided by York Wastewater Consultants of Stamford, CT.

This Plan of Action is based on data obtained from the following sources:

- "Initial Assessment Study (IAS) of the Naval Education and Training Center, Newport, R.I." by Envirodyne Engineers, Inc.
- Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities, EPA/530/SW-611, August, 1977.
- U.S. Army Toxic and Hazardous Materials Agency, Minimum Requirements for Boring Logs, Drilling Procedures and Monitor Well Installation.
- Methods for Chemical Analysis of Water and Wastes, EPA/600/4-79-020.
- Procedures for Handling and Chemical Analysis of Sediment and Water Samples, Technical Report EPA/CE-81-1.
- Standard Methods for the Examination of Water and Wastewater, American Public Health Association, 15th edition, 1980.

In addition to the above documents, this Plan of Action is based on observations made and discussions carried out during a site reconnaissance on October 17 and 18, 1983. Present were:

Loureiro Engineering Associates:

Julio Loureiro	Principal
Charles A. Jaworski	Project Manager
Jeffrey J. Loureiro	Project Engineer

York Wastewater Consultants

Robert Q. Bradley	Laboratory Director

NETC
Martin Dwyer

NORDIV
Thomas Sheckels

This Plan of Action presents a sampling and analysis program which, within the budget limitations of the A/E contract, will provide the most useful data for evaluation of whether or not the six sites being investigated are having an adverse impact on the environment. It is not the purpose of this verification phase of the work to establish the full extent or type of impact on the environment. Further sampling and analysis in the later characterization phase may or may not be necessary at any specific site to more fully define such impacts. The characterization phase may include more extensive sampling of the types described herein with the addition of ground water monitoring wells. A new plan of action will be prepared for the characterization phase after the results of verification analyses are available.

The selection of sampling stations and parameters for laboratory analysis was based on known or suspected hazardous waste constituents which may be present at each site. The types of samples were selected on the basis of environmental importance (e.g., food sources, food chain, ground water) and the possibility that harmful constituents might have an adverse effect.

Sampling stations have been located as close as possible to the potential points of contamination so that the results will be site specific and can be used to evaluate the impact of each site. More extensive distribution of sampling points may be necessary in the characterization stage to establish horizontal and vertical distribution of contaminants and the degree to which the biota has or is being impacted (e.g. sediment and mussel samples further into the Bay).

Because of the heavy pollution load from many sources into Narragansett Bay and the complex estuarine relationships, it was particularly difficult to select control sampling stations. The philosophy used in selecting the control stations was that they should offer similar abiotic factors and should not be

- close to any known point sources of pollution, but should be close enough to the six sites (but outside the direct influence of the sites) under investigation so that biota and sediments collected at the control stations will have been exposed to similar estuarine conditions as those collected close to the six sites. The differences in analytical results between control samples and site specific samples will give a general indication of the environmental impact of the six sites. It is obvious that all samples, including controls, will be subject to Bay pollution loads. By locating the site specific sample stations very close to the respective sites, the highest probability of detecting the potential pollutants from that site will be achieved. By locating the control stations near the six sites, a comparison can be made between the site specific samples and the control samples with similar exposure to Bay pollutants but without direct influence of the six sites. If the control stations were located outside the Bay, or in very different abiotic environments, such comparisons would not be meaningful because important abiotic factors would not be consistent and the level of pollutants detected could not be evaluated against other similar areas of the Bay.

2. SCHEDULE

It is tentatively planned to conduct the sampling program described herein during the week of November 28 to December 2, 1983. If follow-up work is required, it will be carried out the week of December 5, 1983. To the extent possible, the A/E will notify NORDIV and NETC as to the schedule of activities for each day.

The sampling and analysis is expected to be completed by January 30, 1984 and the draft report on the verification step is expected to be completed by February 28, 1984.

SECTION B - PREPARATION FOR THE WORK

1. PRESENTATION OF PLAN OF ACTION AND SAFETY PLAN

The Plan of Action and the Safety Plan will be presented at NETC on November 7, 1983. By that time, it will have received staff review and appropriate modifications will have been made in response to the comments. Following the presentation, final changes to the Plans will be made and detailed mobilization for the sampling program will ensue.

2. MOBILIZATION FOR SAMPLING

Based on the final Plan of Action, the necessary sampling equipment and containers will be assembled and arrangements made for necessary motor vehicles and a boat for marine sampling. All personnel will be briefed on the Safety Plan. A tentative daily plan of sampling activities will be submitted; this schedule will be revised daily depending on progress of the work, weather conditions and any unforeseen problems.

The A/E will engage the services of a sub-contractor to perform drilling and setting of wells at Tank Farm 4. The actual work will proceed as soon as feasible but not before approval of this Plan of Action and issuance of a Dig-Safe Permit.

SECTION C - SAMPLING

1. SITES TO BE INVESTIGATED

Confirmation studies will be conducted for six sites at the Newport Education and Training Center (NETC) in Newport, Rhode Island. The six sites are located as shown on Figure 1 and include the following:

Site No.	Name
1	McAllister Point Landfill
2	Melville North Landfill
7	Tank Farm 1
12	Tank Farm 4
14	Gould Island Disposal Area
17	Gould Island Electroplating Shop

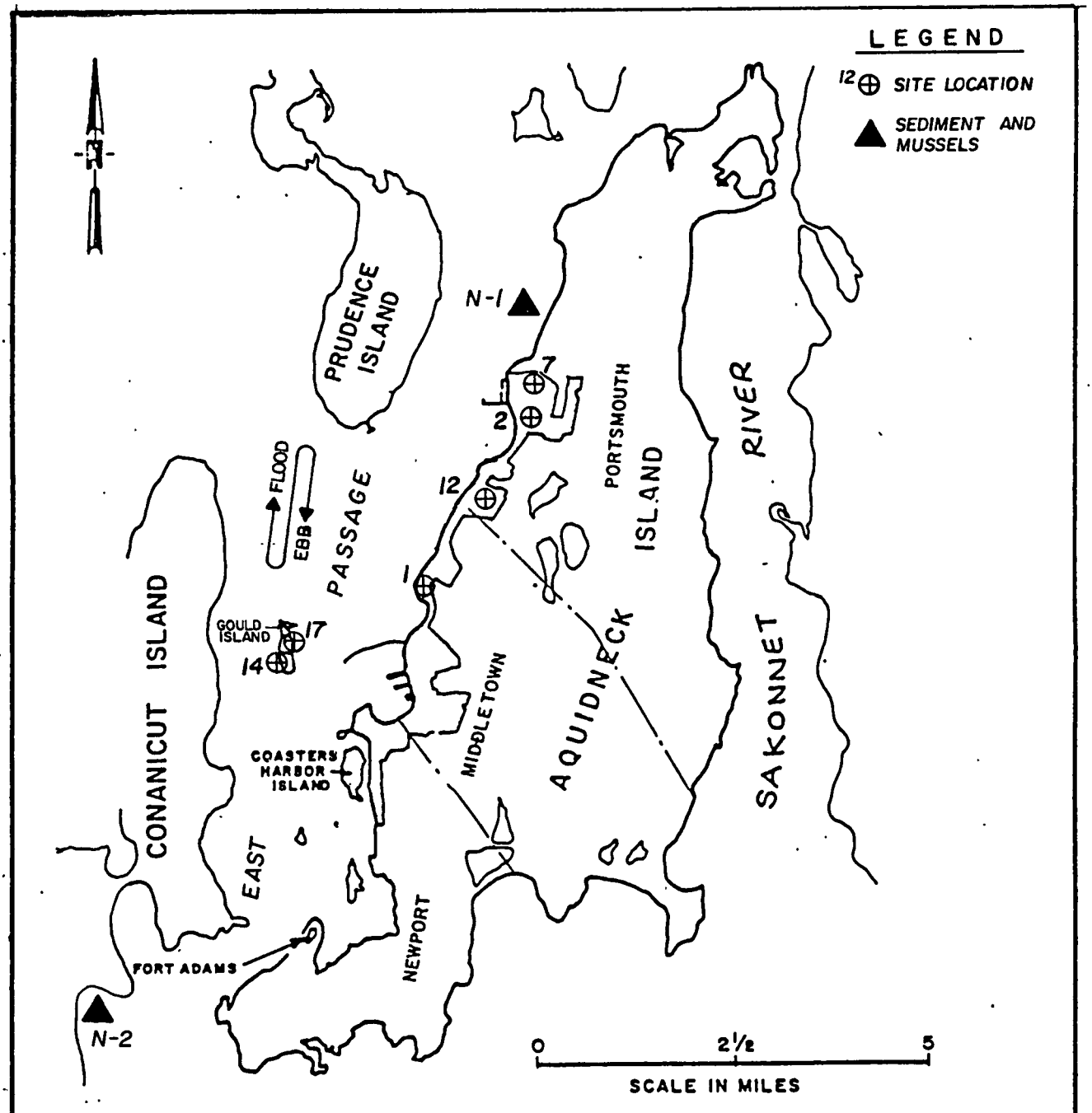
The site numbers are the same as those assigned in the initial assessment study.

2. SAMPLING POINTS

The sampling and analysis program for the verification phase of the confirmation study consists of eight components: sampling of drums suspected to contain hazardous wastes; site specific sampling and analysis at the six sites; and control sampling and analysis. Each of these components are discussed separately in the following sections.

a. McALLISTER POINT LANDFILL

This landfill received all of the wastes generated at the Newport Naval complex from 1955 through the mid-1970's and is known to contain at least 200 gallons of PCB contaminated oil. Also in the landfill are spent acids, waste paints, solvents, and waste oils. Many of the wastes are in direct hydrologic contact with the groundwater and the bay, and surface runoff and leachate seepage from the landfill are directly into the bay. The pathways



NETC SITE LOCATIONS
CONTROL SAMPLING LOCATIONS

YWC
York Wastewater Consultants, Inc.
Stamford, Connecticut

LEA LOUREIRO ENGINEERING ASSOCIATES
a professional corporation
CONSULTING ENGINEERS AVON, CT.

11 - 4 - 83

FIG. N° 1

for pollution migration at this site are readily available, and hazardous wastes are known to be present at the site. If contaminants are entering the Bay, they could adversely affect the biota (shellfish, finfish) which may be harvested for human consumption.

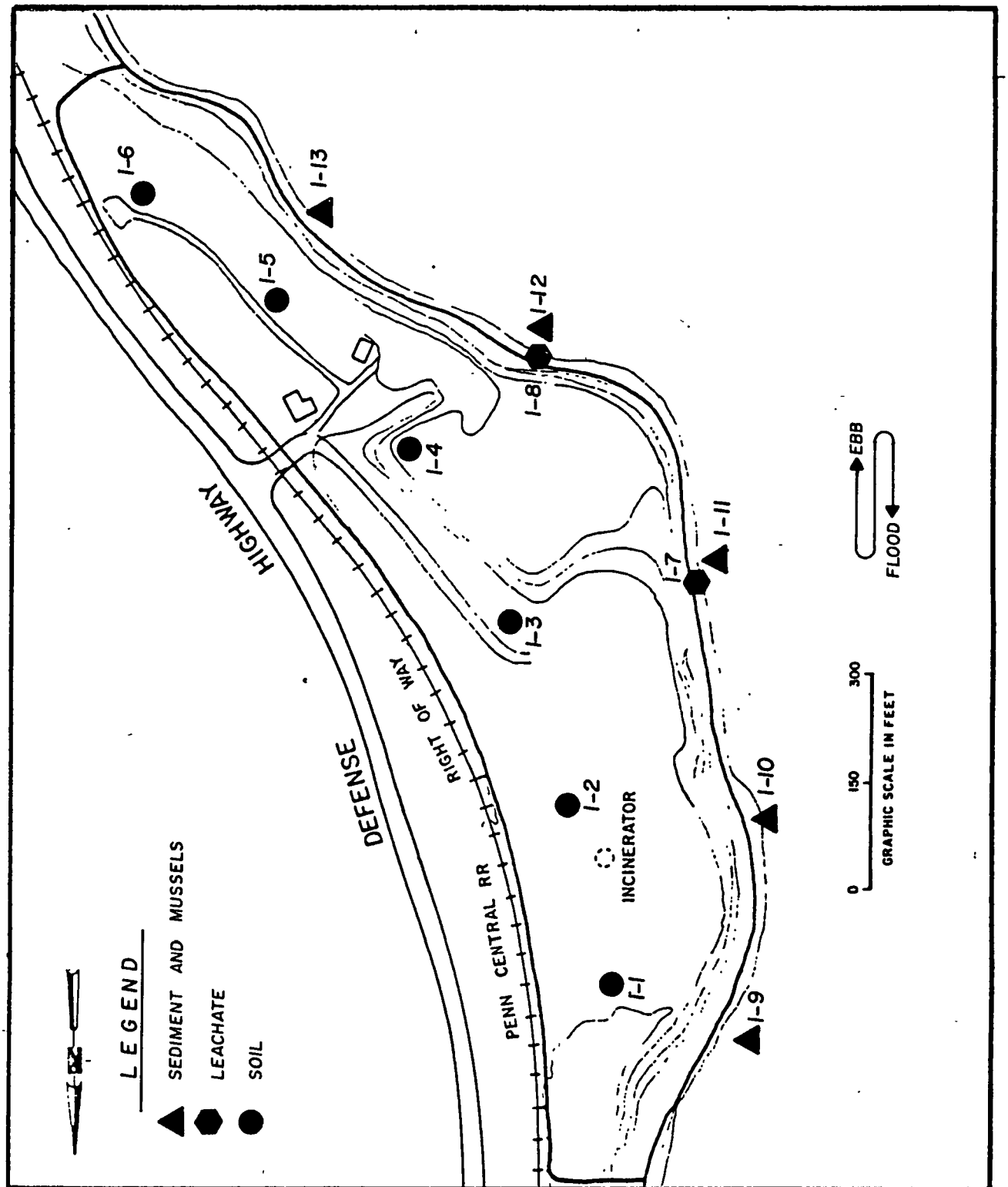
Contaminants in the landfill can enter the surrounding environment by flushing and/or leaching action of (1) natural flow of groundwater toward Narragansett Bay, (2) percolation of precipitation down through the deposited wastes, (3) flow of runoff across exposed waste deposits and (4) movement of tidal flow into and out of the lower part of the landfill. A leachate discharge was observed at one location but the October, 1983 reconnaissance failed to find the second reported leachate discharge.

The sampling and analysis program for this site includes the following parameters: sediment, leachate, mussels and soil. The sampling stations are identified on Figure Number 2. The analyses to be performed are summarized below by sample type:

Sample	Quantity	Analyses
Leachate	3*	Priority Pollutants
Soil	6 stations composited into one sample	Priority Pollutants
Sediment	5	Heavy Metals and PCBs
Mussels	5	Heavy Metals and PCBs

*Wet and dry weather samples will be collected. If the second leachate discharge does not appear during the scheduled sampling period, one of these samples will be deleted.

The types of samples and the parameters of interest have been selected based on data presented in the Initial Assessment Study as well as the chemical and biological properties of the suspected contaminants and the receptors.



NETC SAMPLING LOCATIONS SITE N° 1

McALLISTER POINT LANDFILL

YWC Yost Wastewater Consultants, Inc. Stamford, Connecticut

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11 - 4 - 83

FIG. N° 2

Priority pollutants will be analyzed on leachate grab samples because of the wide variety of materials deposited in the landfill. Priority pollutants will also be conducted on a composite of the soil samples to determine if the surface is contaminated so as to affect growth of vegetation or contaminate runoff. Each of the six soil samples will be taken at locations where vegetation is obviously affected; samples will be taken at depths of 0 to 6 inches and composited to one sample for analysis. If possible, all sediment samples will be taken at a depth of at least two feet below the top of the sediment in water depths of one to ten feet. The top three inches of the core will be analyzed for PCBs and heavy metals (Cr, Cd, Pb, As, Hg, Se, Ag, Cu, Ba, Ni, Be, Sb, Sn). These pollutants are most readily available to the food chain in the top layer of sediment.

Biological samples will consist of mussels collected shoreward of the sediment sampling locations at the lower edge of the intertidal zone. These samples will also be analyzed for PCBs and heavy metals since they are very likely to accumulate in the tissue.

Sediment and mussel sampling points will be collected along the entire length of the landfill.

b. MELVILLE NORTH LANDFILL

This site was used following World War II and up until 1955. Wastes disposed of in this landfill were similar to those discussed for McAllister Point (Site No. 1). This site contains piles of oil saturated soil and tar-like sludge. This site is situated along the shoreline of Narragansett Bay in a low-lying wetland type area. Surface drainage and groundwater flow from the site are directly into the bay, and portions of the site are subject to flooding. Any contaminants dipsosed of at this site would have a

high migration potential. If contaminants are entering the Bay, they could adversely affect the biota (shellfish, finfish) which may be harvested for human consumption.

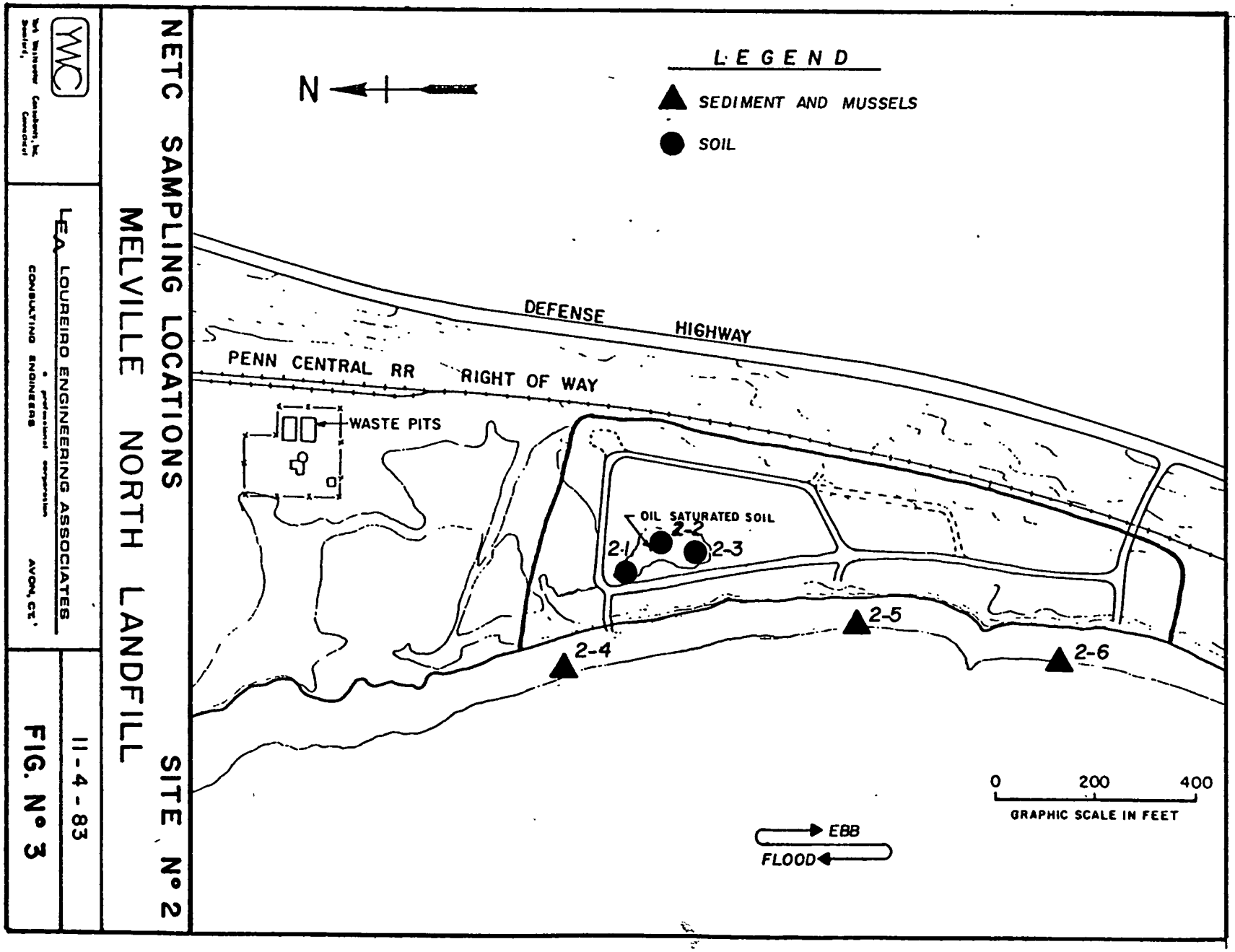
Contaminants in the landfill can enter the surrounding environment by flushing and/or leaching action of (1) natural flow of groundwater toward Narragansett Bay, (2) percolation of precipitation down through the deposited wastes, (3) flow of runoff across exposed waste deposits, and (4) movement of tidal flow into and out of the lower part of the landfill.

The sampling and analysis program for this site includes the following parameters: sediment, mussels and soil. The sampling stations are identified on Figure Number 3. The analyses to be performed are summarized below by sample type:

Sample	Quantity	Analyses
Soil	3 stations composited into one sample	Lead, PCBs and Petroleum based Hydrocarbon
Sediment	3	Heavy Metals and PCBs
Mussels	3	Heavy Metals and PCBs

The types of samples and the parameters of interest have been selected based on data presented in the Initial Assessment Study as well as the chemical and biological properties of the suspected contaminants and the receptors.

Lead, PCBs, and petroleum based hydrocarbons will be analyzed on soil samples to determine if these contaminants are actually present as suspected.


NETC SAMPLING LOCATIONS SITE N° 2
MELVILLE NORTH LANDFILL
LEGEND
SEDIMENT AND MUSSELS
SOIL
N
DEFENSE HIGHWAY
PENN CENTRAL RR RIGHT OF WAY
WASTE PITS
OIL SATURATED SOIL
2-1
2-2
2-3
2-4
2-5
2-6
0 200 400
GRAPHIC SCALE IN FEET
EBB
FLOOD
LEA LOUREIRO ENGINEERING ASSOCIATES
CONSULTING ENGINEERS
AVON, CT.
11-4-83
FIG. N° 3

Each of the three soil samples will be taken at the locations where the suspected oily deposits are visible; samples will be taken at depths of 0 to 6 inches and composited to one sample for analysis. If possible, all sediment samples will be taken to a depth of at least two feet below the top of the sediment in water depths of one to ten feet. The top three inches of the core will be analyzed for PCBs and heavy metals (Cr, Cd, Pb, As, Hg, Se, Ag, Cu, Ba, Ni, Be, Sb, Sn). These pollutants are most readily available to the food chain in the top layer of sediment.

Biological samples will consist of mussels collected shoreward of the sediment sampling locations at the lower edge of the intertidal zone. These samples will also be analyzed for PCBs and heavy metals since they are very likely to accumulate in the tissue.

Sediment and mussel samples will be collected along the entire length of the landfill.

c. TANK FARM 1

Tank Farm 1 is located in Melville North and consists of six underground steel tanks. Each of these tanks has a storage capacity of 60,000 barrels. Five of these tanks are now used for the storage of oils including aviation fuel. One tank is no longer used. In the past, these tanks were periodically cleaned to remove the sludge material which, over time, settles on the bottoms of the tanks. This practice occurred from World War II until the 1970's.

When the tanks were cleaned, the sludge material was placed in a pit which was approximately 20 feet long, 10 feet wide, and 4 feet deep. These disposal pits were simply dug in the general vicinity of the tank being cleaned. The sludge was placed in the pits and allowed to weather for a few weeks. The pits were then covered over and marked with signs warning of

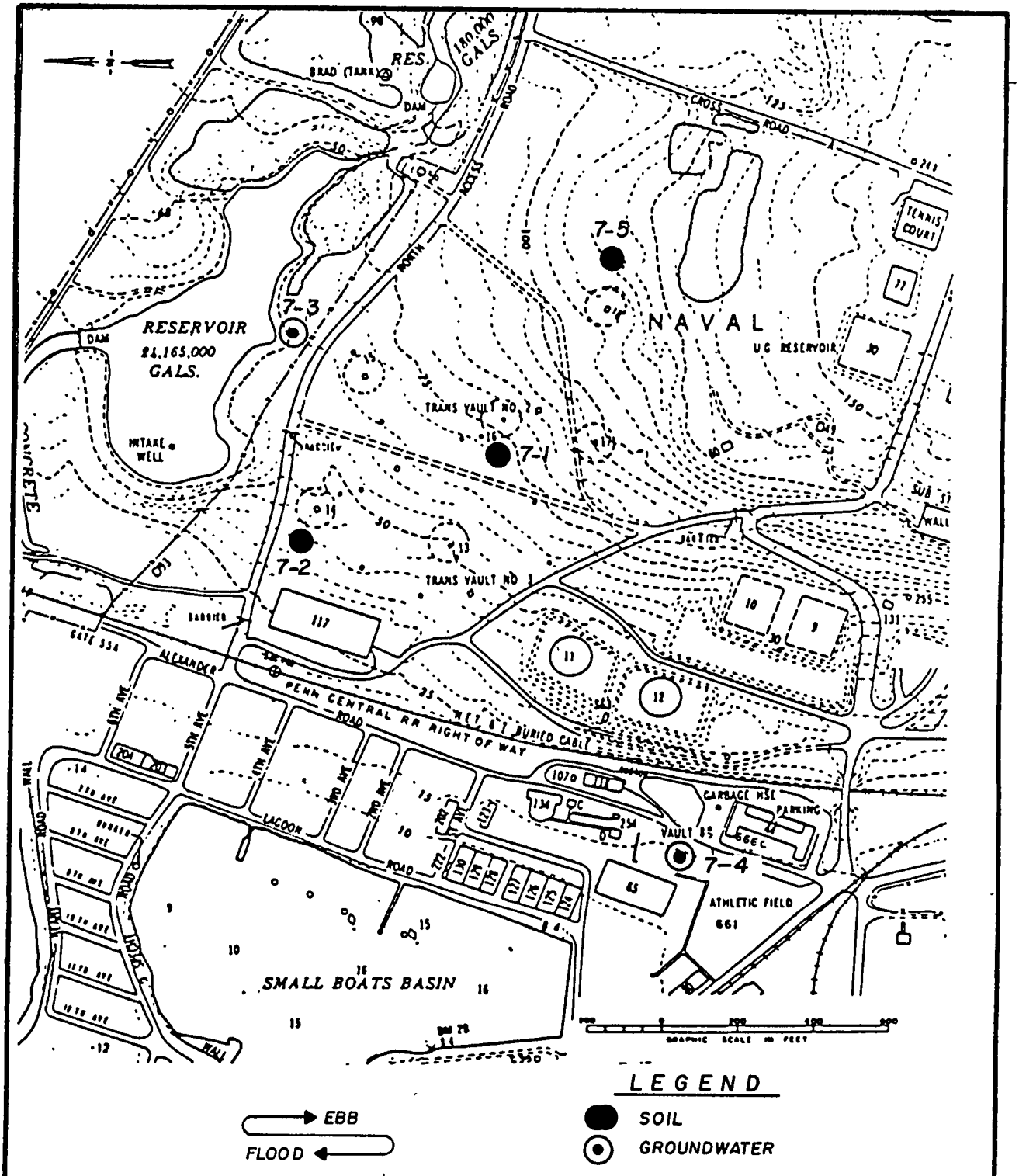
tetraethyl lead. These pits are spread throughout the tank farm, but through the years, most of the signs marking the disposal areas have disappeared. Only two such signs remain at this time.

The potential for contamination does exist at this site. Portions of the tank farm drain northward toward the Melville Pulic Fishing Area, with other areas draining toward Narragansett Bay. The groundwater is generally within 10 feet of the surface. Considering that the waste sludges are in a pit, contaminants would not have to migrate far to reach the groundwater. The bedrock is also very shallow in this area (within 10 or 15 feet). If the bedrock is highly fractured, it would be possible to contaminate the bedrock aquifer. If contaminants are entering the Bay, they could adversely affect the biota (shellfish, finfish) which may be harvested for human consumption. The fish in the Melville Public Fishing area could also be adversely affected.

When the tanks were installed, groundwater drains were placed around each tank. These were individually valved and piped to a common drain. This drain is piped to the west where an oil separator is provided to remove oil if present.

An investigation was conducted by the U.S. Army Corps of Engineers concerning a complaint of oil discharge to the Melville Public Fishing Area. As part of this investigation, a ground water monitoring well was installed near the Melville Public Fishing Area downgradient of Tank Farm 1.

The sampling and analysis program for this site includes the following: soil and groundwater. The sampling stations are identified on Figure Number 4. The analyses to be performed are summarized below by sample type:



NETC SAMPLING LOCATIONS

SITE N° 7

TANK FARM ONE

YWC
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Stamford, Connecticut

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professional corporation
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FIG. N° 4

Sample	Quantity	Analyses
Soil	3	Lead and Oil and Grease
Groundwater	4*	Lead, Petroleum based Hydrocarbons and BTX (Benzene, Toluene and Xylene)

*Includes wet and dry weather samples; two from the existing monitoring well and two from the existing common ground water drain (before the oil separator).

The types of samples and the parameters of interest have been selected based on data presented in the Initial Assessment Study as well as the chemical properties of the suspected contaminants.

Lead and oil and grease analyses will be conducted on two soil samples to determine the presence of these pollutants and potential for migration into the groundwater. These samples will be collected at the two sludge disposal pit locations where the markers are intact plus one other location where such disposal was witnessed. The sampling depth will be three feet.

The common groundwater drain provides a convenient sampling point for determining the presence of contaminants from the tank farm. Analyses will include lead, BTX and petroleum based hydrocarbons. Two samples will be collected at the drain ahead of (or on the bypass around) the oil separator; one sample will be collected during dry weather and one during wet weather.

The existing well near the Melville Public Fishing Area will also be sampled for analysis of lead, BTX and petroleum based hydrocarbons. Two samples will be collected from the well; one sample will be collected during dry weather and one during wet weather.

d. TANK FARM 4

This site has twelve concrete underground tanks, each with a capacity of 60,000 barrels. These tanks were used to store diesel and fuel oil but their use was discontinued several years ago, when they were emptied (but not cleaned) and refilled with water.

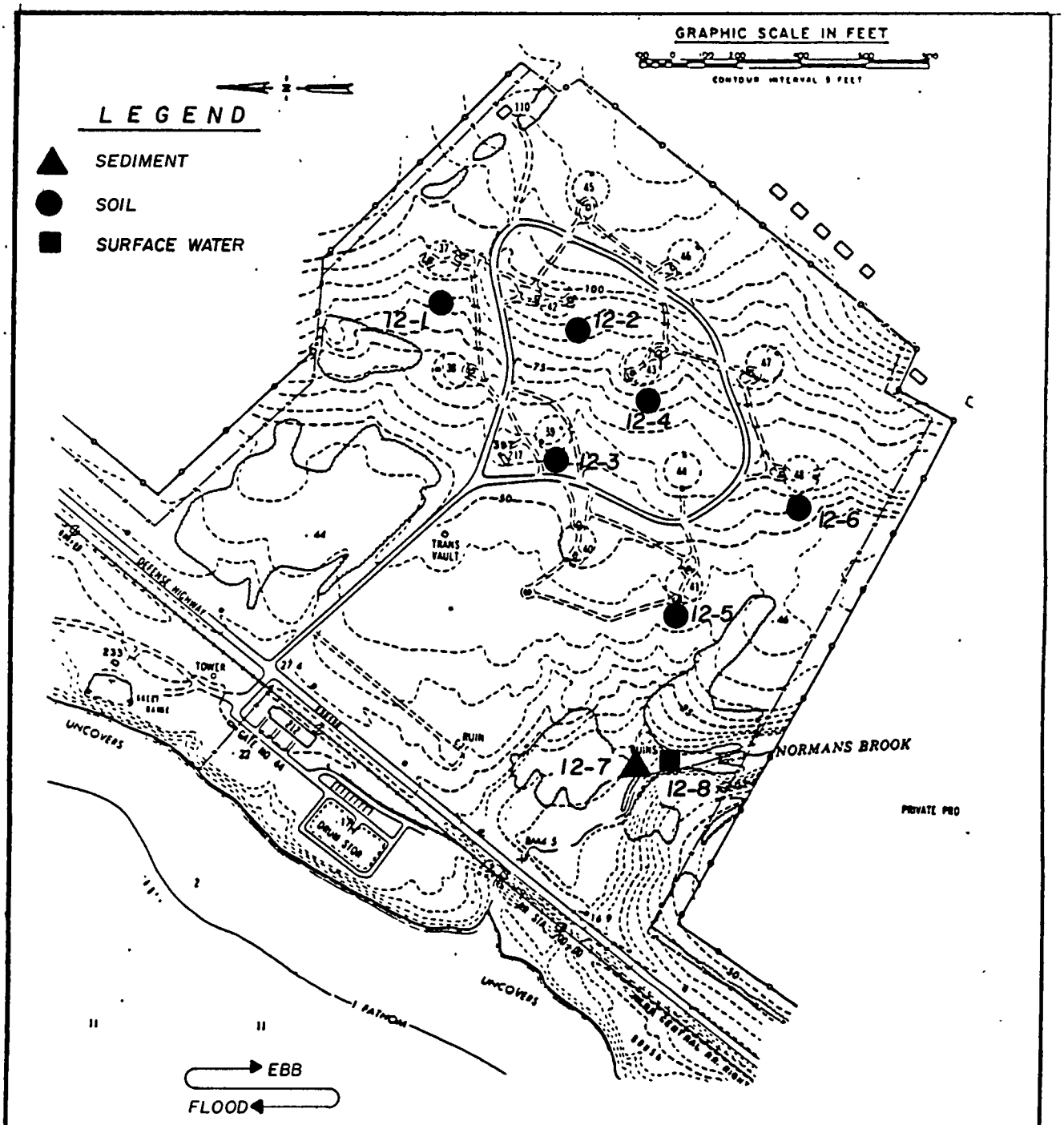
During the period of active use of the tanks, the bottom sludge was periodically removed and disposed of on the ground in the general vicinity of the tank being cleaned.

The potential for environmental contamination exists at this site. Lead and petroleum based hydrocarbons could be transported via surface drainage or could infiltrate down through the soil, where groundwater is very shallow and contaminants would not have to migrate very far vertically to reach the groundwater. This site is located in close proximity to Narragansett Bay. Surface drainage and groundwater flow from these tank farms are into the Bay.

The sampling and analysis program for this site includes the following: groundwater, surface water, soil and sediment. The sampling stations are identified on Figure Number 5. The analyses to be performed are summarized below by sample type:

Sample	Quantity	Analyses
Soil	6 stations composited into one sample	Lead and Oil and Grease
Surface water	2*	Lead and Petroleum based Hydrocarbons
Sediment	1	Lead and Petroleum based Hydrocarbons

*One wet weather and one dry weather sample.



NETC SAMPLING LOCATIONS

SITE N° 12

TANK FARM FOUR

YWC Yost Wastewater Consultants, Inc. Stamford, Connecticut	LEA LOURENSO ENGINEERING ASSOCIATES CONSULTING ENGINEERS AVON, CT.	11-4-83
		FIG. N° 5

The types of samples and the parameters of interest have been selected based on data presented in the Initial Assessment Study as well as the chemical properties of the suspected contaminants.

Lead and oil and grease analyses will be conducted on a composite soil sample to determine the presence of these pollutants and potential for migration into the groundwater. Soil samples will be collected at six suspected sludge disposal locations (on the downhill side of six of the 12 tanks). The sampling depth will be three feet.

Surface water and sediment samples will be collected from the lower end of a swale through which a significant part of the site runoff drains to Norman Brook. The samples will be analyzed for lead and petroleum based hydrocarbons to determine if these pollutants are being released from the site by overland flow. Surface water will be sampled under both wet and dry weather conditions.

New monitoring wells for sampling of ground water will be installed in the characterization stage if necessary.

e. GOULD ISLAND DISPOSAL AREA

This site was the disposal point for incinerator ash and other wastes generated principally during World War II. Also, this site could have received electroplating wastes during World War II. The site is situated along the shore of Narragansett Bay, with many of the wastes coming in contact with the waters of the bay during high tide. Surface drainage and leachate seepage from the site are directly into the bay. The pathways for contaminant migration are readily available at this site. If any hazardous

wastes are transported into the Bay, they could adversely affect the biota (shellfish and finfish) which are harvested for human consumption.

Contaminants in the landfill can enter the surrounding environment by flushing and/or leaching action of (1) natural flow of groundwater toward Narragansett Bay, (2) percolation of precipitation down through the deposited wastes, (3) flow of runoff across exposed wastes deposits, and (4) movement of tidal flow into and out of the lower part of the landfill.

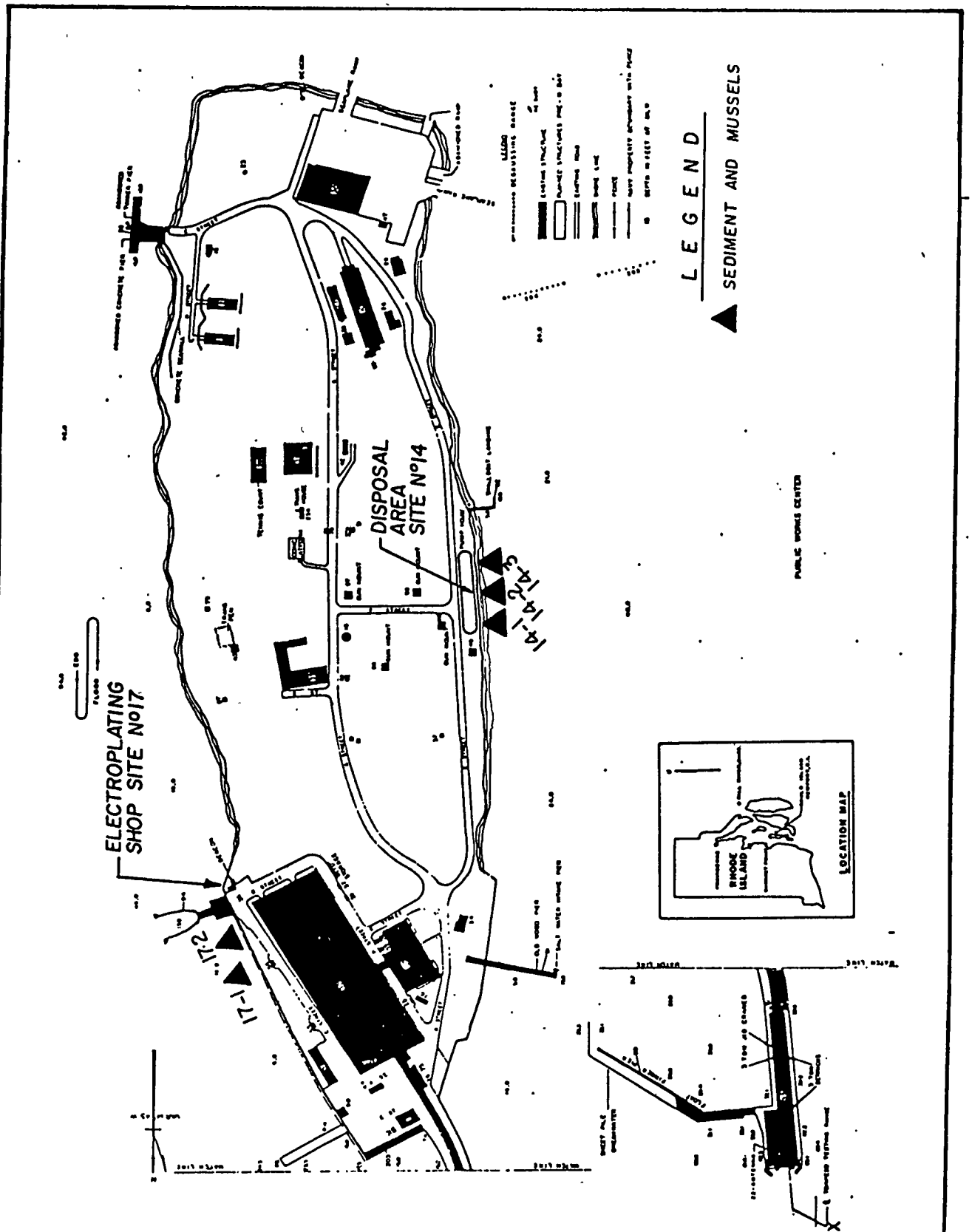
The sampling and analysis program for this site includes the following parameters: sediment and mussels. These sampling stations are identified on Figure Number 6. The analyses to be performed are summarized below by sample type:

Sample	Quantity	Analyses
Sediment	3	Heavy Metals and PCBs
Mussels	3	Heavy Metals and PCBs

The types of samples and the parameters of interest have been selected based on data presented in the Initial Assessment Study as well as the chemical and biological properties of the suspected contaminants and the receptors.

If possible, all sediment samples will be taken to a depth of at least two feet below the top of the sediment in water depths of one to ten feet. The top three inches of the core will be analyzed for PCBs and heavy metals (Cr, Cd, Pb, As, Hg, Se, Ag, Cu, Ba, Ni, Be, Sb, Sn). These pollutants are most readily available to the food chain in the top layer of sediment.

Biological samples will consist of mussels collected shoreward of the sediment sampling locations at the lower edge of the intertidal zone. These



NETC SAMPLING LOCATIONS SITE NOS. 14 & 17

GOULD ISLAND

YWC

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FIG. N° 6

samples will also be analyzed for PCBs and heavy metals since they are very likely to accumulate in the tissue.

Sediment and mussel samples will be collected along the entire length of the landfill.

f. GOULD ISLAND ELECTROPLATING SHOP

Electroplating and degreasing operations wastes were possibly discharged directly into the bay. If these wastes were discharged into the Bay, they could be adversely affecting the biota (shellfish and finfish).

Since these discharges were discontinued several years ago, the principal detectable pollutants remaining in the vicinity of the discharge line will be metals and cyanides in the sediments and, possibly in mussels in the area.

The sampling and analysis program for this site includes the following: sediment and mussels. The sampling stations are shown on Figure Number 6. The analysis to be performed are summarized below by sample type:

Sample	Quantity	Analyses
Sediment	2	Heavy Metals and Cyanide
Mussels	2	Heavy Metals

If possible, all sediment samples will be taken to a depth of at least two feet below the top of the sediment in water depths of one to ten feet. The top three inches of the core will be analyzed for heavy metals (Cd, Cr, Cu, Pb, Hg, Ni, Ag) and cyanide. These pollutants are most readily available to the food chain in the top layer of sediment.

Biological samples will consist of mussels collected shoreward of the lower edge of the intertidal zone. These samples will be analyzed for heavy metals since they are very likely to accumulate in the tissue.

Sediment and mussel sampling points will be concentrated near the discharges lines from the building where the electroplating shop was located.

g. DRUMS SUSPECTED TO CONTAIN HAZAROUS WASTES

Fourteen drums will be sampled to provide sufficient analytical data so that NETC can prepare shipping manifests for off-site disposal of the contents if they are determined to be hazardous wastes as defined in RCRA regulations. The 14 drums are suspected to contain waste oil (7), spent solvent (3), paint sludge (2), sodium hydroxide (1) and transformer oil (1). The analyses to be performed are the RCRA characteristics for the paint sludges and waste solvents; flashpoint and PCBs for the waste oil; RCRA verification of the sodium hydroxide drum; and PCB's on the transformer oil.

All drums will be sampled and the contents composited into five samples to be representative of the five types of wastes.

h. CONTROLS

As discussed in Section A, selection of control sampling locations was complicated by the complex Narragansett Bay estuary and the multiplicity of pollutant discharges to it.

The general location of the control sampling stations are shown on Figure Number 1. These stations were selected because they represent similar habitats, receive similar exposure to pollution loads from the Bay, and yet are beyond the direct influence of the sites under investigation. The analyses to be performed on samples collected at these stations are summarized below by sample type:

Sample	Quantity	Analyses
Sediment	2	Heavy Metals and PCBs + CN
Mussels	2	Heavy Metals and PCBs

If possible, all sediment samples will be taken to a depth of at least two feet in one to ten feet of water. Mussel samples will be collected shoreward of the sediment samples at the lower edge of the intertidal zone.

3. SAMPLE COLLECTION AND HANDLING

a. SAMPLING METHODS

The sampling program at NETC will consist of collecting samples of soil, sediment, groundwater, surface water, mussels, and 55-gallon drums containing suspsected hazardous waste at the locations identified in the previous section. Sample collection techniques for each type of sample are briefly discussed in the following text. Container and sample size, type and preservation requirements are discussed in Section C-3b.

Soil Samples

Soil samples will be collected at depths up to 3 feet. All soil sampling holes will be hand dug to the proper depth and the sample collected in the proper container. The hole will then be refilled with the material excavated. If any materials are encountered which pose a threat to human health or the environment, the digging will be stopped. Sampling will be resumed only if it is determined that this can be done safely.

Sediment Samples

Sediment cores will be taken to depths of at least two feet in 1 to 10 feet of water. The cores will be collected from a boat using an appropriate coring device. Once a core is collected, sediment samples will be placed in the proper containers and the points along the core at which the samples were taken will be recorded. If conditions are such that it becomes impractical to collect sediment cores from the boat, a scuba diver with a hand coring device will be utilized. If there is no sediment at the indicated sample point, a reasonable effort will be made to obtain a sample in another location close to the site under investigation.

Groundwater Samples

The monitoring well at Tank Farm 1 (and other wells planned for the characterization stage) will be sampled using a small peristaltic pump or

manual bailer. The sampling equipment used will be compatible with the analyses to be performed on the sample. All standing water will be evacuated from the well before collecting the sample. If the wells are high yielding, at least three volumes of standing water will be evacuated prior to sample collection.

Surface Water

Surface water samples will be collected using a weighted bottle at a depth of 6 to 12 inches below the surface. The type of container used to collect the sample will be consistent with that required for the analysis to be performed as defined in Section C-3b.

Mussell Samples

Mussel samples will be collected by hand in sufficient number for the analysis to be performed. The samples will be containerized and preserved as discussed in Section C-3b. If there are no mussels at the indicated sample point, a reasonable effort will be made to obtain a sample in another lcoation close to the site under investigation.

55-Gallon Drum Samples

Drum samples will be collected using a Coliwasa type sampling device compatible with the waste to be sampled and the analysis to be performed. The Coliwasa will be thoroughly cleaned between uses.

b. SAMPLE PRESERVATION

The importance of sample preservation between time of collection and time of analysis is a paramount issue. The purpose of the sampling program is to gain knowledge of sample characteristics and identify the source of the sample contamination, if any; any changes in sample composition can invalidate conclusions drawn from data analysis. In other words, results

based upon deteriorated samples negate all efforts (and costs) expended to obtain representative samples.

The ideal way to ensure a lack of sample deterioration is to analyze samples immediately, however this is impractical due to logistic considerations such as:

- the number of samples collected;
- the type of equipment needed for analysis;
- the step involved in sample preparation.

Therefore, some method must be relied upon to extend the integrity of the sample until analysis can be completed. Preservation methods have been documented in the literature for Water and Wastewater (Chemical Analysis of Water & Wastes, EPA, 1979, EPA-600/4-79-020 and 019) and for sediments (Procedures for Handling and Chemical Analysis of Sediment and Water Samples, U.S. EPA, CE-81-1, 1981).

These preservation techniques are intended to retard hydrolysis, reduce oxidation and reduction processes, reduce volatilization and mitigate other undesirable degradation of chemical constituents in the sample. The methods are basically limited in scope to one or a combination of the following techniques:

- pH control;
- chemical addition;
- sample isolation;
- temperature control.

Since different analyses may require different preservation techniques, multiple samples may have to be collected and individually preserved as required, or one can split a sample into subsamples for individual preservation (if the sample is reasonably homogenous in nature).

Sediments

Depending on site location, the parameters to be analyzed on sediment samples at the Newport, RI designated sites include heavy metals (Cr, Cd, Pb, As, Hg, Se, Ag, Cu, Ba, Ni, Be, Sb, Sn), cyanide (total), PCB's and petroleum-based hydrocarbons specifically described site by site elsewhere in this Plan of Action.

The parameters, their related preservation requirements and sample containers are noted in Table 1.

Water Samples

Depending on site location, samples of leachate and ground water will be collected and analyzed for priority pollutants (metals, organics), petroleum-based hydrocarbons and benzene, toluene and xylenes (BTX).

The preservation, container and volume requirements are listed in Table 2.

Soil Samples

Depending on site location, soil samples for the Newport, RI facility site will be analyzed for lead, petroleum-based hydrocarbons, priority pollutants and oil and grease. The preservation, container and volume requirements are shown in Table 3.

Shellfish Samples

Depending on site location, samples of shellfish (mussels) will be taken at the specific locations delineated elsewhere in this Plan of Action. The shellfish will be analyzed for heavy metals and PCB's. The respective volumes, preservation and container requirements are delineated in Table 4.

TABLE 1
SEDIMENT SAMPLE PRESERVATION AND CONTAINER REQUIREMENTS

Parameter	Volume Sample Required	Container Size and Material	Preservation Techniques	Allowed Storage Time
PCB	100 Grams	Wide Mouth 1 Liter Glass with Teflon-Lined Cap (Air Tight)	Cool, 4°C	3 weeks
Petroleum-Based Hydrocarbons	100 Grams	Wide Mouth 2 Liter Glass with Teflon-Lined Cap (Air Tight)	Cool, 4°C	7 days
Heavy Metals	50 Grams	Wide Mouth 1 Liter Glass with Teflon-Lined Cap	Cool, 4°C	6 months
Cyanide	50 Grams	Wide Mouth 1 Liter Glass with Teflon-Lined Cap (Air Tight)	Cool, 4°C	7 days

TABLE 2
*WATER SAMPLE PRESERVATION, VOLUME AND CONTAINER REQUIREMENTS

Parameter	Volume Sample Required	Container Size and Material	Preservation Techniques	Allowed Storage Time
Prority Pollutants:				
- Volatile Organics (includes BTX)	40 ml	50 ml Glass Septum-Capped Vial	Cool, 4°C**	14 days
- Acid Extractables	1 liter	2 Liter Glass with Teflon-Lined Cap	Cool, 4°C	Extract within 7 days, analyzed within 40 days
- Base/Neutrals	1 liter	2 Liter Glass with Teflon-Lined Cap	Cool, 4°C	Extract within 7 days, analyzed within 40 days
- Metals	500 ml	1 Liter Plastic	HNO_3 to pH <2	6 weeks (Hg 2 weeks)
- Cyanide (Total)	500 ml	1 Liter Plastic	Cool, 4°C, NaOH to pH <12	24 hours
- Phenols (Total)	500 ml	2 Liter Glass	$CuSO_4$, H_3PO_4 to pH <4, Cool, 4°C	24 hours
Petroleum-Based Hydrocarbons	1 liter	2 Liter Glass	Cool, 4°C	7 days
Heavy Metals (others)	500 ml	1 Liter Glass	HNO_3 to pH <2	6 months

*Includes leachate and ground water
**Add HCl to pH 1-2 if BTX to be determined.

TABLE 3
SOIL SAMPLES - PRESERVATION, CONTAINER AND VOLUME REQUIREMENTS

Parameter	Container and Size	Volume	Preservation
Lead	Plastic or Glass, 1 qt.	50 g.	Cool, 4°C
Petroleum-Based Hydrocarbons	Glass, 1 qt.	50 g.	Cool, 4°C
Priority Pollutants:			
- Metals	Plastic or Glass, 1 qt.	100 g.	Cool, 4°C
- Cyanide	Plastic or Glass, 1 qt.	100 g.	Cool, 4°C
- Phenols	Glass, 1 qt.	100 g.	Cool, 4°C
- Volatiles	2-Glass, 40 ml. Septum Capped Vial	50 g.	Cool, 4°C
- Acid Extractables	Glass, 1 qt.	100 g.	Cool, 4°C
- Base/Neutral Extractables	Glass, 1 qt.	100 g.	Cool, 4°C
Oil and Grease	Glass, 1 qt.	100 g.	Cool, 4°C

TABLE 4
MUSSEL PRESERVATION, CONTAINERS AND VOLUME REQUIREMENTS

Parameter	Volume Required	Container	Preservation
Heavy Metals	20 grams (20-50 mussels)	Glass, 1 qt. ea.	Cool, 4°C
PCB's	20 grams (20-50 mussels)	Glass, 1 qt. ea.	Cool, 4°C

Drum Samples

At the Newport facility, samples will be withdrawn from fourteen drums and composited as allowed by the Rhode Island Department of Environmental Management. The samples are suspected to be waste oil (7), spent solvent (3), paint sludge (2), sodium hydroxide (1), and transformer oil (1). The analyses to be performed are the RCRA characteristics for the paint sludges and waste solvents; flashpoint and PCB's for the waste oil, verification of the contents of the sodium hydroxide drum and PCBs on the transformer oil. The requirements are to keep the samples in 1 quart glass containers (plastic for the suspected sodium hydroxide) and Cool (4°C) for transportation to the laboratory. The volumes required are 500 grams of each.

c. SAMPLE IDENTIFICATION

Each sample is assigned a sample label which cross references it to chain-of-custody and sampling log records. A three section label is employed which serves the functions of (1) maintaining a seal between sample container lid and body of the container (large portion of label), (2) maintaining chain-of-custody (small portion of label), and (3) affixing to lab request sheet to eliminate numerical transcription errors. The YWC label is shown in the Appendix.

4. SAMPLE CUSTODY

In order to maintain control over the sample from the sampling program thorugh receipt and analysis in the laboratory, a chain-of-custdody program has been instituted for both our convenience and legal considerations.

At the sampling site, the sampling personnel take the sample and transfer it to the appropriate container per Section C-3b. Using the 3-section label (see

Appendix) the large section is affixed on the sample to form a seal between the lid and container. The small section of the label is affixed to the custody sheet (see Appendix) which is then signed off.

Upon arrival of a sample at the laboratory with its Custody Sheet it is transferred to the incoming sample log-in room and the person receiving the sample signs the custody sheet. The sample is then logged in by the Sample Custodian by affixing the long thin section of the York three section label to the sample and log-in sheet in the following fashion. The lower portion of the label (strip) is affixed to the log-in sheet (see Appendix). The sample description is reiterated on this portion of the label. The samll portion of the label is placed on the sample custody sheet in the field.

Each analyst who works on the sample signs the Chain-of-Custody log on the request sheet and maintains responsibility for the sample until the next analyst signs off on it. This procedure is monitored by the Sample Custodian. Upon completion of the analyses, completed results, analyst's intiials, notebook and page numbers are recorded on in-house results sheet (see Appendix) attached to the Request Sheet and given to the Sample Custodian for review. The sample will be stored (or preserved if not already preserved) as dictated by sample type, which is the responsibility of the Sample Custodian. While samples are "work-in-progress" they are stored on the Sample Holding Shelves or the freezer or refrigerator (if required). This is noted on the request sheet for expeditious sample location by the next analyst. Completed samples (excluding water samples) are placed on the thirty day holding shelves and then transferred to the sample storage trailer for a sixty-day holding period. Water and wastewater samples are saved for a two-week period then discarded by the Sample Custodian in a safe and approved manner.

D. LABORATORY ANALYSIS

1. METHODS OF ANALYSIS

Where applicable, all analyses will be conducted in accordance with six (6) major procedural manuals:

(1) Chemical Analysis of Water and Wastes EPA-600/4-79-020, 1979.

(2) Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA-600/4-82-057, 1982.

(3) Procedures for Handling and Chemical Analysis of Sediment and Water Samples, EPA/CE-81-1, 1981.

(4) Test Methods for Evaluating Solid Waste, EPA SW-846, 1980.

(5) Chemistry Laboratory Manual for Bottom Sediments and Elutriate Testing, EPA 905/4-79-014, PB 294 596, 1979.

(6) Standard Methods for the Examination of Water and Wastewater, 15th Edition, 1980.

The basic methods for the related parameters are listed in Table 5.

2. QUALITY CONTROL/ASSURANCE (QA/QC)

QA/QC is performed in accordance with the document in the Appendix of this Plan of Action.

3. REPORTING OF RESULTS

As results are generated they are placed on the raw data results sheet shown in the Appendix. The data is checked by the YWC QA/QC Coordinator, signed off and a report is written and submitted to Loureiro Engineering Associates for interpretation.

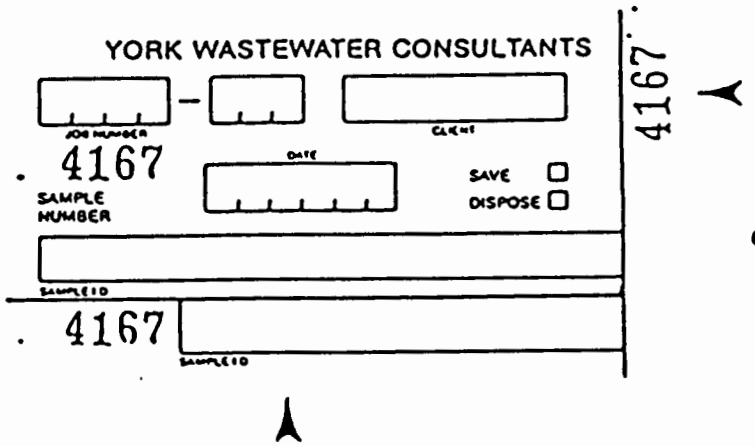
TABLE 5
CHEMICAL ANALYSIS METHODOLOGIES

Parameter	Method Description
PCB's	Extraction followed by concentration and analysis by Gas Chromatography/Electron Capture Detection and/or Gas Chromatography/Mass Spectrometry
Heavy Metals	Acid Digestion followed by conventional flame or flameless Atomic Absorption Spectrometry
Priority Pollutants:	
- Volatile Organics	Purge/Trap/Thermal desorption followed by Gas Chromatography/Mass Spectrometry (GC/MS)
- Acid Extractables	Extraction and concentration followed by GC/MS
- Base/Neutrals	Extraction and concentration followed by GC/MS
- Miscellaneous:	
cyanide (total)	Reflux/distillation/absorption, followed by spectrophotometric analysis
phenols (total)	Distillation followed by spectrophotometric analysis
Petroleum Based Hydrocarbons	Extraction/concentration, followed by GC/MS or extraction followed by infrared spectrophotometric analysis
BTX (Benzene, Toluene, Xylenes)	Purge and Trap/Thermal desorption followed by GC/MS

APPENDIX

LABORATORY FORMS AND SAMPLE LABELS

Affix to sample



Affix to sample custody sheet

Affix to request sheet

YORK

CHAIN OF CUSTODY SAMPLE NO. _____

Location of Sampling: ___ Producer ___ Hauler ___ Disposal Site

___ Other: _____
Sample

Shipper Name: _____

Address: _____
number street city state zip

Collector's Name _____ Telephone: (___)_____
signature

Date Sampled _____ Time Sampled _____ hours _____

Type of Process Producing Waste _____

Field Information _____

_____

_____

Sample Receiver:

1. _____
name and address of organization receiving sample

2. _____

Chain of Possession:

signature	title	inclusive dates
signature	title	inclusive dates
signature	title	inclusive dates
signature	title	inclusive dates
signature	title	inclusive dates
signature	title	inclusive dates

YORK
Laboratories
Division of Y.W.C., Inc.

LABORATORY SERVICES REQUEST

Date Opened: _____ Job No. _____

Client: _____

☐ Lump Sum
☐ T&M

Y.W.C. Division: ALD _____ WW _____ OTHERS _____ Quote: $ _____

Logged by: _____

Sample Number & Type	Parameters to be Determined	Chain of Custody
		Received by Date
		Received by Date
		Received by Date
		Received by Date
		Final Disposition

COMMENTS

Duplicates Required ☐

Status: Rush _____ Routine _____

YORK

RESULTS OF ANALYSES

Project No._____

Client_____

PARAMETER						Analysts Initials	Date Analyzed	Notebook/page#	Checked by

APPENDIX

QUALITY CONTROL/ASSURANCE

YORK

QUALITY CONTROL/ASSURANCE PROGRAM

FOR LABORATORY ANALYSES

1.0 INTRODUCTION

Our business as an environmental consulting laboratory involves providing qualitative and quantitative data to a variety of clients who utilize these data for decision making. To be valuable, the data must accurately describe the characteristics and concentrations of constituents in the samples submitted. In many cases, because these data may impact significantly on process, legal and cost decisions, approximate or incorrect results are in most instances worse than reporting no result at all.

Most analysts practice QC to some degree as a matter of professional pride. However, whether this "some degree" is enough or too much cannot be a subjective decision. To relieve the necessity of our staff from developing personalized QC approaches, the following routine QC program was prepared by laboratory management and addresses the following items:

- Personnel Responsibilities for the QA/QC program:

    a. Vice President/Division Project Manager
    b. Chief Chemist/Quality Control Coordinator
    c. Laboratory Manager/Analysis Coordinator
    d. Associate Chemist/Sample Custodian

- Chain of Custody Procedures:

    a. Sample Log-in
    b. Sample History
    c. Sample Retain
    d. Sample Disposal

- General Laboratory Techniques:

    a. Sample Integrity
    b. Glassware Preparation
    c. Reagent Preparation
    d. Sample Considerations
    e. Sample Handling
    f. Notebook Format
    g. Reporting Results
    h. Other Considerations

- Specific Laboratory Techniques (Instrumental):

  a. Atomic Absorption Spectrophotometry
  b. Total Organic Carbon Analysis
  c. pH Measurement
  d. Conductivity
  e. UV-Vis Spectrophotometry
  f. Bomb Calorimetry
  g. Elemental Analysis (C,H,N)
  h. Gas Chromatography
  i. Infrared Spectrophotometry
  j. Gas Chromatography/Mass Spectrometry
  k. Analytical Balances
  l. Aquametry (Karl-Fischer)

- Specific Laboratory Techniques (Physical and Wet Chemistry):

  a. Viscosity
  b. Density, Specific Gravity
  c. Color, Odor
  d. Flash, Fire Points
  e. Pour Point
  f. Titrimetric Analyses
  g. Gravimetric Analyses

The analytical methods used in our laboratory conform to the following criteria:

- The selected methods should measure the desired constituents of the samples in the presence of normal interferences with sufficient accuracy and precision to meet the client's needs.

- The selected procedures should utilize the skills and equipment we possess.

- The selected methods have been sufficiently tested to have established their validity.

- The methods are practical and sufficiently rapid to permit routine use if required.

- The methods are theoretically sound, all factors considered, and can be practically applied to various sample types and are referenced in the available literature.

On a routine basis, most of our analytical work is described in the following documents:

1. Standard Methods for the Examination of Water and Wastewater (USPHS), 15th Edition, 1980

2. Chemical Analysis of Water and Wastes (EPA-600/4-79-020, 1979)

3. Chemical Analyses of Bottom Sediments and Elutriation Testing (EPA)

4. The Federal Register

5. Analysis of Pesticides in Human and Environmental Samples (EPA)

6. American Society for Testing and Materials (ASTM) Methods

7. American Society of Mechanical Engineers (ASME) Power Test Codes

8. Association of Offical Analytical Chemists (AOAC) Methods

9. Scott's Standard Methods for Chemical Analysis

10. United States Pharmacopeiea Methods Manual

11. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA-600/4-82-057, July, 1982

12. Test Methods for Evaluating Solid Waste, U.S. EPA SW-846 and SW-846B, 1980

13. Procedures for Handling and Chemical Analysis of Sediment and Water Samples, EPA/CE-81-1, May, 1981

The specific QA/QC references utilized by the laboratory and which served as a basis for this program are listed as follows:

- Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA 600/4-79-019, March, 1979.

- Manual of Analytical Quality Control for Pesticides in Human and Environmental Media, EPA 600/1-76-017, February, 1976.

- Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA-600/4-82-057, July 1982.

2.0 PERSONNEL RESPONSIBILITIES

Independent of the magnitude of a project, a team is designated to address the proper performance of QA/QC. Generally this team is comprised of four individuals who share the various related duties. In the York organization the individuals (by job title) and their QA/QC roles are noted in the following table.

Table 1
QA/QC Role Delineations

Title	QA/QC Role
Vice President, Laboratory Director	Division Project Manager
Chief Chemist	Quality Control/Assurance Coordinator
Laboratory Manager	Analysis Coordinator
Associate Chemist	Sample Custodian

The specific duties of each individual of the QA/QC team are described in the following paragraphs.

Laboratory Division Project Manager (P.M.)/Vice President

The responsibility of the Division Vice President/P.M. is to control and assure the quality of all Laboratory data generated. Specific duties to comply with these responsibilities include:

- Preparation of project specific work plan which includes analytical and QA/QC Methods to be utilized.
- Direct and coordinate with the Chief Chemist/QA/QC Coordinator on work plan implementation.
- Coordinate with and direct the Laboratory Manager/Analysis Coordinator regarding logistics such as personnel, instrumentation and supplies for scheduling purposes and that properly trained personnel are available.

- Coordinate with and direct the Associate Chemist/Sample Custodian to ensure sample integrity considerations to include chain-of-custody, sample log-in, tracking of samples through analyses, receipt of all data and presentation of data to the Chief Chemist/QA-QC coordinator for result verification.

- Develop external QA monitoring protocol through the use of "blind" samples prepared by the U.S. EPA for water/wastewater and hazardous waste analysis.

Chief Chemist/QA/QC Coordinator

The QA/QC Coordinator's responsibilities include implementation, enforcement and monitoring of all QA/QC procedures. In addition, the following duties are also the responsibility of the QA/QC Coordinator.

- Coordinate with the P.M. in the development of the QA work plan including numbers and types of blanks, spikes, replicates.

- Certify that each analyst is thoroughly trained and "checked out" on applicable procedures.

- Maintain and inspect instrumentation logs to include calibration and performance records.

- Review QC data to verify analyses results are within acceptable limits; construct and maintain control charts for each parameter to monitor system problems.

- Coordinate with the sampling personnel or Engineering Project Manager to determine the appropriate sampling equipment and containers.

- Ensure that all sample containers destined for project use are prepared for sampling to eliminate contamination.

- Prepare field spikes, blanks, duplicates and QC spikes (in-house) as called for in the work program.

- Verify that all sample containers are charged with proper preservatives (where applicable) and ensure that holding times and shipping considerations (e.g. coolers, 4°C) are addressed.

Laboratory Manager/Analysis Coordinator

The Analysis Coordinator has full charge of the Laboratory operation regarding logistics and has the following related responsibilities.

- Coordinate with the Division P.M. and QA/QC Coordinator regarding the Analytical plan and ensure that all QC measures are executed.

- Ensure that all chemists and technicians assigned to the project are properly trained as evidenced through performance checks.

- Verify that procedural consideratons as outlined in the specific work program are met (i.e.: GC/MS conditions, etc.).

- Follow-up procedural verification through the program to ensure that initial compliance with work program elements is carried through.

- Assess results for data presentation clarity, consistency, accuracy and precision and determine if reruns are required.

Associate Chemist/Sample Custodian

The Laboratory Sample Custodian is responsible for sample receipt, chain-of-custody compliance, data handling and cursory review and sample storage/disposal. The specific duties of this position are as follows:

- Verification that received samples correspond to the chain-of-custody transfer section of the Request Sheet.

- Inspection of the samples with particular regard to sample condition, preservation requirement maintenance or other considerations which may impact on the analytical result.

- Verify that all documentation on the samples (labels) is correct, properly attached and that the requested analysis parameters are consistent with the Work Program. This latter step requires interface with the Analysis Coordinator who may further interface with the Engineering Project manager if required - and then assigns the respective analyses.

- Maintain all records to ensure that chain-of-custody is maintained from receipt to storage/disposal of samples.

- Prepare the data generated in coordination with the Analysis Coordinator for presentation to the Division P.M. and QA/QC Coordinator for review.

- Ensure that client dictated sample storage times are met. Where applicable, ensure that proper disposal of samples is performed including drum or lab-pack storage for disposal by a licensed commercial disposal firm.

3.0 CHAIN OF CUSTODY/SAMPLE LOG-IN

In order to maintain control over sample flow from receipt to analyses in the laboratory, a chain-of-custody program has been instituted for both our convenience and legal considerations.

Upon arrival of a sample at the laboratory with its Custody Sheet (see Fig. 1) it is transferred to the incoming sample log-in room. The sample is then logged in by the Sample Custodian by affixing the long thin section of the York three section label to the sample and log-in sheet in the following fashion. Referring to Figures 2 and 3 (label and log-in sheet respectively), the lower portion of the label (strip) is affixed to the log-in sheet. The sample description is reiterated on this portion of the label. The largest portion of the label is filled in with the job number, date, disposition instructions, and comments as required. The small portion of the label is placed on the sample custody sheet in the field.

Each analyst who works on the sample signs the Chain-of-Custody log on the request sheet and maintains responsibility for the sample until the next analyst signs off on it. This procedure is monitored by the Sample Custodian. Upon completion of the analyses, completed results, analyst's initials, notebook and page numbers are recorded on in-house results sheet (See Figure 4) attached to the Request Sheet and given to the Sample Custodian for review. The sample should be stored (or preserved if not already preserved) as dictated by sample type, which is the responsibility of the Sample Custodian. While samples are "work-in-progress" they are stored on the Sample Holding Shelves or the freezer or refrigerator (if required). This is noted on the request sheet for expeditious sample location by the next analyst. Completed samples (excluding water samples) are placed on the thirty day holding shelves and then transferred to the sample storage trailer for a sixty-day holding period. Water and Wastewater samples are saved for a two-week period then discarded by the Sample Custodian in a safe and approved manner.

3.1 Sample History

An important facet of analytical chemistry involves a cursory understanding of the sample origin. Intimately related to this

YORK

CHAIN OF CUSTODY SAMPLE NO. _____

Location of Sampling: ___ Producer ___ Hauler ___ Disposal Site

___ Other: _____
Sample

Shipper Name: _____

Address: _____
number street city state zip

Collector's Name _____ Telephone: (___)_____
signature

Date Sampled _____ Time Sampled _____ hours _____

Type of Process Producing Waste _____

Field Information _____

_____

_____

Sample Receiver:

1. _____
name and address of organization receiving sample

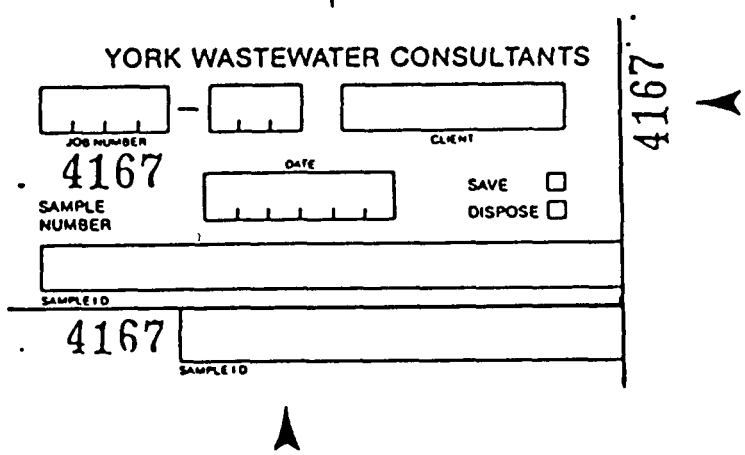
2. _____

Chain of Possession:

signature	title	inclusive dates
signature	title	inclusive dates
signature	title	inclusive dates
signature	title	inclusive dates
signature	title	inclusive dates
signature	title	inclusive dates

fig. 1

Affix to sample



Affix to sample custody sheet

Affix to request sheet

YORK
Laboratories
Division of Y.W.C., Inc.

LABORATORY SERVICES REQUEST

Date Opened: _____ Job No. _____

Client: _____

Y.W.C. Division: ALD _____ WW _____ OTHERS _____

☐ Lump Sum
☐ T&M

Quote: $ _____

Logged by: _____

Sample Number & Type	Parameters to be Determined	Chain of Custody
		_____ Received by Date
		_____ Received by Date
		_____ Received by Date
		_____ Received by Date
		_____ Received by Date
		_____ Final Disposition

COMMENTS

Duplicates Required ☐

Status: Rush _____ Routine _____

YORK

RESULTS OF ANALYSES

Project No. _____

Client _____

PARAMETER						Analysts Initials	Date Analyzed	Notebook/page#	Checked by

fig.4

is knowledge of sample constituents., Some knowledge of these two preliminary facts will typically make an analysis much easier and lend more confidence to the result. The knowledge of what a sample contains is very important when quantitative analysis is sought. Major and minor constituents in a sample which may cause interferences in the analysis are germain to correct analysis. The point is that one should always ask questions regarding sample history to address proper sample preparation and analysis. This eliminates reruns and produces analytically acceptable data on a first-time-through basis.

3.2 Sample Retain

Due to the instability of many of the sample types we analyze, samples are kept on the holding shelf or refrigerator for a period of two weeks. At this point, aqueous samples are discarded and other samples are stored in the sample storage trailer for an additional sixty days. At this point, the samples are discarded in an environmentally acceptable manner by the Sample Custodian.

4.0 GENERAL LABORATORY TECHNIQUES

Integral to successful analysis is good laboratory technique. In turn, good technique is strongly rooted in the preliminary steps of analysis preparation. Sample integrity, glassware preparation, reagent preparation, sample considerations and handling, and results reporting are all prerequisite parameters for proper analysis.

4.1 Sample Integrity

The worth of an analytical result to a client is directly related to the confidence one has in the sample itself regarding representativeness, changes due to volatilization, instability, photodecomposition, etc. To minimize these stability concerns, samples should be preserved, where applicable, utilizing standard techniques (water and wastewater). In any case, if unstable materials are suspected, refrigeration, deep-freezing, or other techniques are applied.

Regarding representativeness, it is our duty as a consulting laboratory to be sure the samples are representive enough for regulatory scrutinizing, client considerations or other applications. In cases where clients provide samples to us, we must touch base with the client regarding his satisfaction that the samples we are analyzing are representative for his ultimate use of the data we generate.

4.2 Glassware Preparation

One of the most critical aspects of a laboratory analysis is the cleaning of glassware so that no contamination of the sample occurs. A few basic principles should be borne in mind when cleaning glassware.

Initially all glassware should be rinsed with water, or appropriate solvent (i.e. toluene or hexane in the case of oil samples) and placed on a dirty glassware cart. This will save much time and energy later. Most glassware should be cleaned with warm soapy water. Markings can easily be removed by scrubbing or using acetone. After cleaning in soap, the glassware should be rinsed with tap water, then twice with labora-

tory water. This procedure will suffice for most of the routine glassware used in the laboratory.

Glassware for metal analyses, including volumetric glassware, should be rinsed with 1+1 acid (hydrochloric or nitric) followed by rinsing with laboratory water prior to use. This will suffice for all metal analyses except mercury.

Glassware for mercury must be doubly rinsed in 1+1 nitric acid followed by triplicate rinsing with laboratory water. This includes any and all glassware used in the digestion and analysis.

Certain other types of analyses require special glassware cleaning. Glassware used for pesticide or PCB analysis must be Chromerge® treated, dried and rinsed with nanograde solvent (hexane or petroleum ether) prior to use. Muffling at 400°C is also an acceptable technique, but sometimes impractical considering the size of the glassware used.

Glassware for volatile organic analyses should be rinsed with methanol and the dried at 105°C for at least 30 minutes.

Keep in mind that the purpose of cleaning glassware is to eliminate a source of error in your analysis and hence is an important aspect of the analysis worth the time and effort.

4.3 Reagent Preparation

Proper reagent preparation is germain to attaining good results. If the reagents are prepared wrong, the desired reaction will probably not take place. This will result in a worthless analysis and cause a waste of one's valuable time.

Reagents should be made up according to common sense. Certain short cuts are allowable in some cases, but not in others. In any case, consult related literature.

Standard and stock solutions are the most critical and are worthy of a little extra time and consideration. Use a laboratory balance and be careful with what you are doing. Write

weight values down in your notebook for future reference. Do not use scrap paper.

Most indicators do not require exact weights or volumes. (Exceptions are redox indicators like ferroin used in the COD analysis). Whenever a direction states so many grams per liter can also be made up using the graduations on a beaker or directly in the appropriate container, do not waste time using volumetric glassware for these types of solutions.

All reagents should be stored in an appropriate container, not a volumetric flask. The container should be well marked with the reagent name, concentration, initials of whoever made it up, and the date. Keep this in mind beforehand and plan time to prepare the containers as your solutions are dissolving. Put reagents back on the appropriate shelf (not on the bench) after you are done with the analysis. Don't leave clutter around for someone else to clean up.

4.4 Sample Considerations

The size of a sample needed for a given analysis depends upon what and how much of it one expects to find. Initially the sample should be as homogeneous as possible. Liquids are generally not a problem, but solids usually demand grinding with a mortar and pestle. Any knowledge of sample history is helpful.

The volume of a liquid one takes for a water analysis should be the sample size recommended in Standard Methods unless sample history indicates low or high levels which might require deviations from normal volumes. Other exceptions are made when the total sample volume is small compared to the volume needed for all analyses requested. In this case a smaller sample size is usually dictated. The use of common sense is often helpful in determining what sample size is needed.

4.5 Sample Handling

Samples, whether they be water, sludge, soils or solids reach the laboratory in various conditions. Handling them in the proper fashion to keep their chemical integrity is important.

Most samples that we receive require refrigeration to prevent losses due to degredation. Certain parameters require preservation to remain stable. Samples that reach our doors from outside sources that are not properly preserved should be done by us as soon as we receive them. A list of routine parameters listing preservatives, holding times and sample volumes needed is listed in the EPA Chemical Analysis of Water and Wastes Manual. Everyone should be aware of these and see to it that analyses are run on the properly preserved samples.

Attention should be made to the amount of sample submitted for analysis. External clients and Engineering Project Managers are instructed to submit more than enough sample to allow for possible double checks on analyses that seem questionable or for further analysis. Every analyst should be aware of sample volume present before preceeding with preparation. Thought should be taken of the amount of sample available, amount you need for your particular analysis, and analyses remaining to be performed before your aliquot is taken. This will assure that enough sample will be available for all, as well as extra sample for rechecking or additional analysis. This is important because many times the sample submitted is all that is available, if more sample is needed, the analyses already run are meaningless because the sample concentration of pollutants can change at the source and we will end up comparing results of two different samples.

As stated before, much of the work we receive requires refrigeration. There are two refrigerators for this purpose. One refrigerator shall be designated to hold samples of work in progress. The other will be for work not yet started, holding of samples per client's future instructions, and retaining samples in which analyses are complete. Once a job is finished, the samples should be removed from the "work in progress" refrigerator and placed into to the holding refrigerator. This is the responsibility of the Analyst. Water samples are to be held for two weeks after report is mailed in the "holding" refrigerator in case the client has a question or requests further analysis. If no further work is required, the Sample Custodian can then discard the sample and bottle. In this fashion there is a constant flow of sample containers from refrigerator to refrigerator to disposal.

It is the Sample Custodian's responsibility through Analyst coordination to see to it that after use, samples are put back in the proper refrigerator. This should be done right after an aliquot is taken, to keep the sample at the proper storage temperature.

4.6 Notebook Format

Every consulting laboratory provides data to clients which has potential legal implications. For this reason and as a matter of good record keeping bound, sequentially numbered page, carbon-copy notebooks are used. The day's work should be legibly presented in the notebook and follow a logical presentation pattern suitable for review by others. In essence, good notebook format is complete and allows another scientist to easily follow a progression from the beginning of the analysis to the final result. For example, samples of soil are submitted for lead analysis. The notebook format should contain, as a minimum, the following key elements:

- date
- job number
- client name
- abstract or name of method
- tabular presentation of data to include York number and all raw data

For the example, analysis of lead in soils, a copy of proper notebook format is attached on the following page. Any corrections on the data should be initialed. If IR, GC, GC/MS or Flameless AAS is the technique utilized, the scans which are generated are filed appropriately with a notation in the notebook as to where the scans are filed.

4.7 Reporting Results

Report all results on the appropriate result sheet as soon as the analysis is completed. Report analyses with the appropriate number of significant figures.

For example, a suspended solids sample was found to contain 10 mg per 100 ml. A graduate cylinder (or wide mouth pipette) was used for the sample and a five place analytical balance was used for the weighing. The accuracy of the balance was $\pm$

3/14/83
01-6043-06 John Doe Company

Request - Lead (Pb) in soil samples taken by YWC

method - digestion of 5g. samples (±0.01mg) in HNO_3 / H_2O_2, filtration, AAS

Sample #	Dry basis Sample wt (g.) gross - tare = net g.	final Volume (mls)	mg/l from AA	tot. mgs	(ug/g) ppm
4363	21.16135 - 16.16258 = 4.99877	100 ml	0.81	0.081	16.20
4364	22.16483 - 17.12345 = 4.99877 g.		0.80	0.08	~~16.00~~ 16.00
4365	21.16100 - 16.16000 = 5.00100		1.06	0.106	21.2
4366	21.18000 - 16.18000 = 5.00000		3.04	0.304	60.8
4367	21.13332 - 16.13213 = 5.00119		4.06	0.406	81.18
BLANK	N/A	100 ml	<0.5	<0.05	

BB

Calculation:

$$ug/g\ Pb = \frac{mg/l\ (\text{from AA}) \times \text{final vol.}\ (l) \times 1000}{\text{wt. (g.)}}$$

(tot. mgs)

0.00005 g (or ± 0.05 mg) while the graduate cylinder is ± 0.5 mls. The error associated with two weighings and two volume readings would be ± 1.00 mg/l. The final value would then be 100 ± 1.00 mg/l. The reported value, therefore, should not have any decimal places.

If a 500 ml sample was taken (instead of a 100 ml sample) and the weight found was 10 mg/500 mls the associated error would be 20 ± 0.14 mg/l and therefore one decimal place would be allowed. Unless a large sample volume was taken (>250 mls) do not report any decimal places.

4.8 Other Considerations

(A) Desiccators

We have many desiccators in the balance room. As with refrigerators, each desiccator should be designated for specific purposes, (i.e. work in progress, glassware and crucibles prepped and conditioned for use, work that is to be held, and dried chemicals to be used for standard stock solutions). This will avoid confusion as to what can be taken for use and what should not. Once a crucible or piece of glassware is weighed back, it should be removed from the balance room, cleaned and prepared for the next time it is needed or placed in the holding desiccator, depending on the type of analysis performed. Again, any equipment put in the holding desiccator, should only remain there for two weeks after the final report has been mailed to the client. A good practice is to check this desiccator at the end of every week to be sure it is up to date.

One person is assigned to see to it that the dessicant is not spent. A schedule is set up to make sure there is always dry dessicant in the desiccators at all times to assure no sample has absorbed moisture. This is accomplished by always having dessicant drying in the drying oven and changing to newly dried dessicant first thing Monday morning, sooner than once a week if deemed necessary.

(B) Water Quality

Many analyses and reagent preparation require the use of deionized/distilled water. The quality of this water which we generate is monitored weekly for pH and conductivity; monthly for volatile organics, heavy metals, chlorine and standard plate count and yearly for bacteria and priority pollutants..

In addition, in all required analyses, a blank comprised of water and reagents (where applicable) is run to correct for any reagent or water contribution.

(C) Log-Books

As a mechanism for tracking instrument and incubator performance, log books are provided for each. Upon use (or other established frequency) pertinent parameters are recorded. Periodically, log books are examined and any significant change in a critical parameter triggers further examination. This is basically an operational tool intertwined with preventive maintenance.

5.0 SPECIFIC LABORATORY TECHNIQUES (INSTRUMENTAL)

The need for good instrumental analysis is paramount in a laboratory such as ours. With state-of-the art instrumentation the tendency is to emphatically trust the microprocessor systems for QC purposes. In fact, good QC is required on a more regular basis for instrumentation than with wet chemistry since the thought processes are minimized and are handled by electronic means. Should a malfunction occur, the only monitor is a good QC program.

5.1 Atomic Absorption Spectrophotometry (AAS)

Atomic absorption spectroscopy is a very useful instrumental technique for metals analysis. Basically, the technique involves absorption of radiation specific to an element by aspirating the sample into a flame which is aligned with a beam of radiation emitted from a lamp containing the element in question. The amount of radiation absorbed is proportional to the concentration of the metal in the solution being aspirated. A more detailed theoretical discussion can be obtained by consulting the "cookbooks" and the various instrumental analysis books in our library.

When using the AAS it is important to insure the burner head angles and flame conditions are optimized. Failure to do so could result in both poor sensitivity and a very narrow linear working range.

This optimization is performed in the following manner:

- Light the flame and optimize the absorbance reading for a high (as listed in the "cookbook") standard by adjusting the flame condition.
- Continue aspirating the standard and adjust the burner height to achieve maximum absorbance.
- Adjust the burner angle to achieve the maximum absorbance reading.

The AA is standardized using working range standards which are prepared from commercially prepared 1000 ppm stock certified standards. The stability of the working range is verified on use of the instrument by preparation of one standard in the working range. This standard is aspirated along with the working standards and samples and it's result is reported in lab notebooks versus the working range standard. This serves as a monitor of degradation of the working range standard. If the results for the fresh single standard and the working standard differ by ± 5%, the working range standards must be prepared fresh.

When analyzing samples, the sample readout must be bracketed by working standards. If dilution, burner head angle adjustment or further sample concentration is necessary to achieve bracketing then one or more of these steps must be performed.

Prepare a blank acid and calibration standard using the same type of acid as will be used for sample processing. The calibration standards should cover the range of the samples, where this is known or can be reasonably determined. Aspirate the blank, followed by the standards from lowest to highest concentration. A minimum of three aspirations (flame analysis) of each blank and standard will be performed to establish the calibration has not changed, blanks and standards will be run not less frequently than after every 20 samples, but more frequently if in the judgement of the analyst a system change seems likely. Flameless analysis generally requires more frequent calibration (after evey 10 to 15 samples).

If the sample matrix is so complex that viscosity, surface tension, and components cannot be accurately matched with standards, the method of standard additions will be used. Standard additions will be made to portions of the sample, and the calibration curve will be prepared from the responses to these aspirations. These standards will be analyzed in the same manner and frequency as the previously mentioned reagent standards. This standard additions technique is utilized as Standard Operating Procedures on flameless (graphite furnace) Atomic Absorption.

For each element a description of interferences and control of these interferences is listed in the Perkin-Elmer Methods Manual. Typical methods for control of matrix interferences or ionization involve addition of Lithium or Lanthanum, altering flame conditions and/or flame fuel gas or utilizing an alternate wavelength. It is each analysts job to investigate these potential problems as delineated in the methods manual.

With each set of samples run, a QC standard prepared by the QC coordinator is run and the recoveries are noted in the QC log. A variance of ± 10% from the actual result of the QC standard will trigger a more detailed examination of the technique.

In addition, one sample out of each lot is prepared and run in duplicate to monitor the analytical precision. This data is also reported in the QC log.

5.2 Total Organic Carbon Analysis

The TOC analyzer is set up and calibrated with a 180 ppm carbon standard in accordance with the manufacturers instructions. The 180 ppm standard is run in duplicate to determine the precision. The reading should be 180 ppm ± 2 ppm or ± 3% whichever is greater.

Subsequent to this, an organic-free water is injected in duplicate to determine that the system is working properly. Each sample is run in duplicate. Should a sample result in a high TOC value (>1000) a blank should be run before the next sample to verify system stability.

If the working range of the samples is 1-20 ppm, a standard of 18 ppm (or 9 ppm) should be run to verify the linearity of the system.

5.3 pH Measurement

The measurement of pH is a measurement of the hydrogen ion activity in a given aqueous solution. The measurement of pH in non-aqueous sytems has no real meaning. The pH measurement is dependent upon the correct response of the electrodes. Clogged, dirty or fouled electrodes will give poor response and may lead to inaccurate results.

Electrodes should be cleaned by soaking in warm acid (1:1 HNO_3 to remove organics and warm 1:10 HCl to remove inorganics) and then well rinsed in laboratory water. Insure that the tip of the reference electrode is not clogged with precipitated salts; the electrode is filled with KCl solution, and the opening at the top of the electrode is open so as not to form a vapor lock.

Standardization of the Meter

1. Set the slope to 100 and put the electrodes in buffer solution. Let sit several minutes to equilibrate and then adjust calibration know to set pH at buffer value. (pH = 7.00)

2. Rinse electrodes and place in laboratory water. Put electrodes in the second buffer (pH = 10.00) and after several minutes set readout to buffer value using the slope control.

3. Rinse electrodes and put back in laboratory water. Unit is now ready for use.

Sample pH Determination

1. For routine samples simply place electrodes in solution and with occasional stirring wait for reading to equilibrate. (Sample should be at ambient temperature)

2. Report pH to two decimal places. For low ionic strength solutions e.g. (distilled waters or boiler waters) add a few drops of KCl solution to the sample before reading.

3. Rinse electrodes and place in laboratory water between readings.

5.4 Conductivity

The measurement of conductivity is a measure of how well a solution will conduct electricity and hence its dissolved salt concentration. The unit of conductivity is micromhos/cm.

Standardization

1. Put the electrode in a 1880 μmhos/cm standard and turn the unit on set at Scale 4. Adjust the standarization control to achieve the desired value.

2. Rinse the electrode and place it in laboratory water. The unit is now ready for use.

Samples

1. Put the electrode in solution. Set meter at Scale 5 and turn it on.

2. Adjust the scale setting until the reading is greater than 20% of full scale. Agitate the solution to achieve equilibrium and record the reading.

3. Turn the unit off, set meter to Scale 5 and rinse the electrode. Put the electrode back in laboratory water and go on to the next sample.

Note: If the sample has a high pH add Gallic acid to overcome hydroxide ion interference. If a given sample is off scale it may be diluted and the reading multiplied by the dilution factor.

5.5 UV-Visible Spectrophotometry (Colorimetry)

Colorimetric analysis is a basic technique in our laboratory. The methodology is based on Beer's law which basically states that the absorbance of a species in solution at a certain wavelength is proportional to its concentration, the path length through which the beam of light travels, and a constant called the molar absorption. In other words the intensity of the color of a soluble substance in solution is related to its concentration. The intensity of the color is called the absorbance and it is measured at a given wavelength using a spectrophotometer.

In general it is important to exactly follow the procedures and conditions outlined in Standard Methods in order to achieve the

desired results, namely determining the accurate concentration of the species in question. Also, the cells used in the analysis should be scrupulously clean so that no foreign matter affects the absorbance readings. Make certain that the wavelength as well as the pathlength are correct so that the conditions under which the standard curve was set up are duplicated.

A master set of standard curves for routine colorimetric analyses have been prepared and are in the Chief Chemist's office.

Each time a colorimetric analysis is run, a minimum of two standards and a reagent blank are run to verify the method. If the standards are greater than + 10% off the master calibration curve, a new calibration curve is prepared.

Should the sample absorbance exceed the calibrated range, it must be diluted to fall within the linear working range of the particular method. In addition, with each lot of samples a QC standard prepared by the QC coordinator is submitted and analyzed concurrently. A variance of more than 10% will trigger a detailed look at analytical technique.

5.6 Bomb Calorimetry

This technique is utilized to determine the Heat of Combustion of a material and can also be used to determine certain elemental concentrations such as sulfur, chlorine and other halogens.

For Heat of Combustion determination, the oxygen bomb is calibrated with benzoic acid. This procedure is done on a monthly basis (or if the instrument is not used for a long period of time, it is done on use). Samples are weighed to 0.1 mg and one out of each lot is analyzed in duplicate to monitor analytical precision. Other quality control techniques are delineated in the ASTM methods manual.

5.7 Elemental Analysis

Carbon, Hydrogen and Nitrogen analyses are performed on the Perkin-Elmer Model 240 analyzer. This instrument requires only a 1-3 mg sample, therefore one should be confident that the

portion to be analyzed is representative of the total sample. For solids it is recommended that the sample is ground in a mortar and pestle and sieved through a 44μ screen.

The instrument must be thoroughly stabilized as indicated by reproducibility of the ladle blanks. Normally 4 ladle blanks are run followed by 2 additional runs which are typically reproducible. Subsequent to blanks, an acetanilide standard is run in duplicate to establish the instrument sensitivity factors. Experience has shown that subsequent to initial blanks and standardization the following sequence should be utilized - run 3 samples followed by a standard then repeat the sequence.

5.8 Gas Chromatography (GC) (Basic, Packed Column)

Most every chromatographer has developed a "unique" approach to analyses requiring GC. Some basic rules that insure reasonable quality control are listed as follows:

(A) Injection Technique; in our laboratory we utilize: 10 μl syringes graduated in 0.2 μl units. Typically 1 to 5 μl of sample is injected into the column depending upon such things as detector type, column type, concentration, etc. The most accurate injection technique and the technique we utilize is a modified "solvent-flush" technique. Basically, for a 1.0 μl injection you withdraw 1.0 μl of sample, wipe the syringe needle with a Kim-Wipe® and then withdraw 1.0 μl of air. You will notice the volume of your sample is not 1.0 μl, but approximately 1.6 μl (This is due to the volume of the needle.) However, when you depress the plunger for injection only 1.0 μl will be delivered. The syringe is inserted into a septum and immediately depressed, held for a 5 second count then quickly removed.

When using a syringe for dilution of standards or samples be aware that once you've injected your concentrate into the dilution medium do not withdraw the diluted sample as a means of mixing or cleaning the syringe. This will result in a higher final concentration than you expected since you've also added the 0.6 μl from the syringe needle.

(B) Column Selection; a wealth of literature and experience in our laboratory is at your disposal to help you choose the proper column for a GC analysis. The column chosen should be able to separate the compounds of interest efficiently. If no literature is available for your specific problems (this will be rare) keep in mind some rules of thumb:

- What types of compunds you're working with are important - is it a highly polar material or very non-polar (n-aliphatics); is it reactive or thermally labile?

- The major types of liquid phases for packed column use are classified as very polar, moderately polar, low polarity and non-polar. These types should correspond to the polarity of your material.

(C) Chromatographic Conditions; as stated earlier, the literature lists various chromatographic conditions to effect good analysis.

In general, the injection port and detector temperatures should be equal or the detector temperature slightly warmer. The injection port should never be significantly (10°C) hotter than the detector as condensation on the detector may occur.

One should also keep in mind the compounds to be chromatographed. Many times injection port temperatures if too hot can decompose or rearrange certain compounds which are thermally labile.

(D) Quantitative Analysis; in our laboratory the peak height or peak area concepts are used. As long as the standards and samples are run under the exact same chromatographic conditions this method is accurate, allowing for matrix affects which are examined through surrogate checks.

The following guidelines are used for quantitative analysis and are listed chronologically:

1. A blank is run to show that the system is interference free. This blank is run at a high sensitivity. For example, for electron capture detection, the blank is run at x 8 or less; for FID the blank is run at x 4 or less. These attenuations are applicable to isothermal situations where a clean system is usually the case since there should be very little baseline drift caused by column bleed.

   When temperature programming with high liquid phase loadings (>5%), some column bleed may create baseline drift necessitating lowering the sensitivity to some level.

2. A series of standards is prepared which cover the lower detectable limit you are going to achieve, and two (2) other ranges which are dependent upon where you think your sample will fall.

   These standards are injected going from the lowest to the highest concentrations.

3. Subsequent to these injections, the sensitivity factor (S_f) for each standard is calculated. They must agree within ± 10% or a problem exists. This problem may be detector linearity, injection technique or thermal decomposition of the material in the injection port, to name a few.

   The S_f is calculated by the following equation:

$$S_f = \frac{\text{Standard Concentration}}{\text{peak ht. (mm) x attenuation}}$$

   or

$$S_f = \frac{\text{Standard Concentration}}{\text{Area}}$$

Peak areas are measured using electronic integration.

Peak heights are measured in accordance with the following figures:

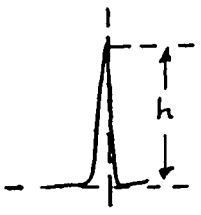


Figure 1

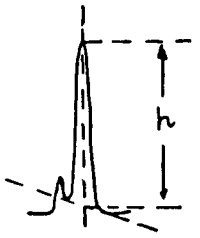


Figure 2

Figure 3

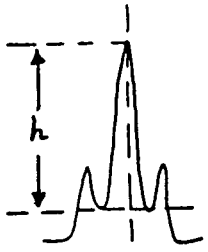


Figure 4

Different references show different ways of measuring peak heights, however, the rule of thumb is that the measurement technique used is not critical as long as the technique to measure standard and sample is identical, and no matrix affects are apparent.

4. At all times the peak height or area of a sample must be bracketed by two standards, one higher and one lower, preferably on the same attenuation scale. This will insure the sample height is within the linear working range of the particular column/detector system.

The linear working ranges of the GC detectors we have at York are listed as follows:

Detector	Working Range	Comment
Flame Ionization	0.5 ppm to 10,000 ppm	Depends upon type of compound
Thermal Conductivity	0.1% to 100%	Independent of type
Electron Capture	0.1 ppm to 100 ppm	Only for electron deficient atoms (O,N,Cl,F,S)
Flame Photometric	0.1 ppm to 100 ppm	Only for sulfur or phosphorus compounds

5. Qualitative Analysis; GC per se is not a definitive qualitative tool. The combination of retention time, detector type and column type is sufficient to make only a preliminary qualitative assignment. Many compounds may co-elute and GC offers no method for deconvolution of coeluting compounds. Even dual column confirmation is not definitive evidence. Only GC/IR or GC/MS techniques are strictly applicable to the task of definitive identifications.

In any case, one should spike the sample with a known standard and examine the scan to insure the analyte peak height increases.

5.8 Gas Chromatography/Mass Spectrometry

Each day the GC/MS system is evaluated in terms of the chromatography and mass spectrometry. The chromatography (capillary columns only) is evaluated by injection of a Grob-type mixture and methane into the GC/MS. The resulting scan is used to measure column performance parameters such as capacity (K'), theoretical plates (N) and average linear velocity (u).

The Mass Spectrometer system is Autotuned (Hewlett-Packard System) with perfluorotributylamine (PFTBA). The computer then normalizes the spectrum of PFBTA to that of the EPA recommended standard Decafluorotriphenylphosphine (DFTPP). The overall GC/MS performance is then checked by injection of a 500 ppm standard of DFTPP in methanol to pass the following criteria.

Reference Compound Key Ions and Ion Abundance Criteria

Mass	Low Abundance Criteria
51	30-60% of mass 198
68	Less than 2% of mass 69
70	Less than 2% of mass 69
127	40-60% of mass 198
197	Less than 1% of mass 198
198	Base peak, 100% relative abundance
199	5-8% of mass 198
275	10-30% of mass 198
365	1% of mass 198
441	Less than mass 443
442	Greater than 40% of mass 198
443	17-23% of mass 442

The major use of the GC/MS in our laboratory is for identification analysis of volatile priority pollutants and the Base/Neutral and Acidic organic fractions from extraction of wastewater, air samples and hazardous waste samples. The related QC techniques are as follows.

5.8.1 Volatile Organics

Samples to be analyzed for the EPA designated volatiles are taken using metal or teflon bailers (if a well is sampled) and transferred to 40 ml teflon septum capped glass vials. The vials are prepared by washing with Liquinox® soap solution, rinsing with water and drying at 103°C a minimum of 4 hours. The vials are stored in the 103°C oven until use.

When sampling, the vials are filled in such a manner that no air bubbles are allowed to flow through the sample. The sample is allowed to overflow the bottle and the cap is applied. Two (2) vials are taken at each location.

The vials are inverted to check for air bubbles. If any air bubbles are detected, the vial is refilled. The vials are immediately sealed and placed in a cooler (4°C) until analyzed. The analysis is performed within 7 days of sampling according to EPA Method 624 (Purge & Trap/Gas Chromatography/Mass Spectrometry Method). A field blank is carried along with the samples to monitor any cross contamination.

The analytical quality control is performed according to the following protocol.

Standards for the GC/MS System are purchased from Supelco, Inc., Bellafonte, PA. These stock standards are certified and are then used for serial dilution to achieve working range standards which are prepared daily.

A calibration curve for each volatile and surrogate is prepared initially and covers the range 1 ug/l to 400 ug/l. If samples fall outside the range, the sample is diluted to bring it within the working range.

Each day the curve is verified using one standard. If significant drift (>2 standard deviations) has occurred, a new curve is constructed.

Each day the system is conditioned with an organic-free water blank and demonstrated to be interference-free.

Each sample has the following quality control related to it.

The sampling precision is verified by analyzing the two field replicates. The analytical precision is determined by analyzing one sample in duplicate.

Since the sample matrix can affect the individual recoveries of certain volatiles, a surrogate standard consisting of bromochloromethane, 2-bromo-1-chloropropane and 1,4 dichlorobutane is injected into one out of every five samples and the recovery is reported.

In addition, for each ten samples submitted, a "blind" quality assurance sample prepared by the Chief Chemist is submitted for analysis and the respective recoveries are noted.

The Mass Spectrometry System is autotuned and computer normalized to the EPA criteria for decafluorotriphenyl phosphine (DFTPP).

The qualitative analysis of a particular peak is performed by obtaining an Extracted Ion Current Profile (EICP) for the primary ion and two other ions as listed in the EPA 624 Method.

In addition, the mass spectrum is plotted and library searched to provide ancillary confirmation. The quantitative analysis is performed by using the EICP for the primary ion for quantitation as defined in EPA 624.

5.8.2 Base/Neutrals, Acids and Pesticides/PCB's

Samples of water to be analyzed for Base/Neutrals, Acids and Pesticides/PAC's are withdrawn from a groundwater monitoring well using teflon tubing. Samples are taken in duplicate. No

tygon or other potential sources of contamination are included in the sampling system. The sample containers are 1 liter or 1 gallon amber glass with teflon lined caps (or aluminum foil if sample is not corrosive). These bottles are washed with detergent, rinsed with distilled water and rinsed thoroughly with acetone followed by methylene chloride.

Once samples are transferred to these bottles they are transferred to coolers (4°C) and brought to the laboratory. Within 48 hours from sampling, the samples are extracted. If this is impractical due to the man-power availability or other considerations, the samples are preserved by adding 35 mg of sodium thiosulfate per each ppm of residual chlorine (if present) in the sample; adjust the pH to 7-10 using sodium hydroxide or sulfuric acid and the volume of each respective preservative added is recorded.

Subsequent to preservation all samples are extracted within 7 days of collection and analyzed as soon as possible thereafter (but within thirty days of collection).

Before extracting any samples, organic-free water is processed in the same manner as a sample. This method blank establishes the degree to which the reagent/extraction/concentration/analytical systems are interference free. In addition to a method blank, a field blank is maintained with the samples.

Sampling precision is assured through analysis of a field duplicate. Analytical precision is determined through analysis of one sample in duplicate. In addition, the samples are spiked with surrogate standards as follows: for base/neutrals we use anthracene-d_{10}; for acidic compounds we use phenol-d_5. These surrogates establish the recovery of the method and are injected into each sample.

An initial calibration curve is developed for each compound covering a range which will give a final concentration in the sample of 1 ppb through 1000 ppb (2 ng and 2000 ng) (assuming 1.0 ml final volume and a 2.0 µl injection). Any samples falling outside of this working range are diluted accordingly. Each day the analyses are run, one standard is injected and compared to the initial calibration curve. If no more than two

standard deviations are found, no further calibration is performed. If greater than two standard deviations are noted, a new curve is constructed.

For each ten samples submitted a "blind" quality assurance sample is prepared by the Chief Chemist and submitted. This QA technique is used to determine accuracy of the method.

The mass spectrometry system is autotuned and spectra are normalized to the EPA Standard DFTPP. Each day the base/neutrals and phenols are measured, the column performance is verified using benzidine and pentachlorophenol respectively.

The system is operated in the Extracted Ion Current Profile mode in which each peak is monitored for the three characteristic ions listed in EPA Method 625. In addition, the mass spectrum of each peak is plotted and library search is performed by computer as ancillary confirmation.

5.9 Infrared Analysis

Infrared (I.R.) analysis is based upon the absorption of infrared radiation at various wavelengths by bonds between atoms. Examples are the C=O absorption at around 1650 cm^{-1} and the O-H absorption around 3200 cm^{-1}. Compounds, especially organic compounds, each have individual I.R. spectrum due to the absorption of I.R. radiation at various wavelengths. Comparison of the I.R. spectrum of an unknown to various known spectra can be used to identify what class of compounds the unknown belongs to and even identify the unknown material.

Usually the most important aspect of good I.R. analysis is the preparation step. This involves preparing a KBR pellet, a thin film on NaCl or AgCl plates, or using the mull technique. The KBR pellet technique involves grinding the sample with I.R. grade KBR (10 parts KBR to 1 part sample), placing the powder in a pellet press, and forming a KBR "window". The "window' should be thin enough to see through yet cover the entire opening of the press.

The thin film method calls for putting a few drops of the material between two salt plates and running a spectra. Solu-

tions of materials can also be run in this manner using solution plates. A blank is also run to correct for the solvent used.

The mull technique uses two types of oil, halocarbon oil and mineral oil (also called Nujol). A portion of the sample is ground with each material and two spectra are run. The halocarbon oil samples is run from 1400 - 4000 cm^{-1} while the mineral oil sample is run from 750 to 1500 cm^{-1}. This technique is useful for insoluble materials.

The IR performance is monitored prior to use by running a spectrum of reference polystyrene.

5.9.1 Analytical Balances

The most important piece of equipment in any analytical laboratory is the analytical balance. The degree of accuracy of the balance is ultimately reflected in gravimetric analysis and all weight prepared standards.

The balances are maintained under a yearly contract which maintains the balances traceable to NBS standards.

In addition, the balances are to be checked on a bimonthly basis against Class S weights and the balance performance noted in the log books.

5.9.2 Karl-Fischer Aquametry

The Karl-Fischer (K.F.) technique is used to determine water in a variety of materials. The sample is mixed with a solvent, usually methanol or a glycol and titrated using the K.F. titrator. The K.F. apparatus is initially standardized against water (weighed out from a syringe) and the value of the reagent expressed in mg water per ml reagent.

A sample of the unknown is weighed out and titrated. The weight of water is then calculated and the concentration determined.

Several types of materials can cause interferences. It is therefore best to check the literature prior to analyzing a new type of sample.

6.0 SPECIFIC LABORATORY TECHNIQUES (PHYSICAL METHODS)

The determination of pour point, flash point, fire point, density, and viscosity are all based directly on the appropriate A.S.T.M. Methods. It is necessary to follow these procedures very closely so that duplicate results can be obtained between individuals and laboratories. This is especially true of the heating/cooling rates mentioned in the flash, fire, and pour points. Too rapid a rate will yield false information on the sample and incorrect results. Be sure that density and gravity results are corrected to the appropriate temperature.

7.0 SPECIFIC LABORATORY TECHNIQUES (WET CHEMISTRY)

A variety of samples we analyze on a day to day basis require some type of wet chemistry. Typically this involves either a gravimetric step or a titrimetric step.

7.1 Gravimetric Analysis

Gravimetric analysis involves the weighing of some substance in order to determine its concentration. Examples are the sulfate and ash determinations in fossil fuel samples. Generally a gravimetric determination involves precipitation of an insoluble salt (e.g. Barium sulfate) from solution, filtering the salt and isolation of the salt in order to weigh it. The isolation step usually is a drying technique of some sort (at 100°C or higher).

Other compounds require rather sophisticated techniques for precipitation such as the use of dimethylglygoxime for nickel or palladium determinations. The point is that one should check the literature for the necessary conditions, temperatures, etc. to insure that:

- The salt is entirely precipitated;
- The salt has the desired composition;
- The salt is easily isolated from solution.

Isolation of the salt usually involves filtration. Most of the time filter paper or Gooch crucibles are used depending upon the properties of the salt (e.g. crystal size, drying temperature, etc.). Thorough washing of the precipitate is usually necessary in order to remove contaminants which would lead to erroneous results. Care must be taken not to lose any of the desired precipitate during the washing step.

Drying of a precipitate in a Gooch crucible requires placing the crucible in an oven at 105°C-110°C for 1-2 hours, cooling in a dessicator and weighing.

Ashing a filter paper involves careful folding of the paper and placing it in a tared crucible. The paper is slowly charred (no flame should occur) and then gradually heated to at least

600°C to remove any traces of carbon. The crucible is allowed to cool in a desiccator and weighed.

From the weight of the precipitate the desired substance can then be quantified.

7.2 Titrimetric Analyses

The titration technique is one of the most widely used quantitative methods. A number of applications are available:

- Acid/Base Titration
- Precipitation Formation
- Complex Formation
- Oxidation-Reduction

In our laboratory these four category types of titrations are routinely performed.

The QC procedures are listed along with the references in Standard Methods and ASTM and should be adhered to.

8.0 ANALYST CERTIFICATION

The proficiency of all analysts is demonstrated through internal check samples. The Analysis Coordinator prepares two (minimum) unknowns which are submitted to the analyst in duplicate. Each sample is analyzed and the results used to verify that the analyst can produce accurate and precise data. Once accuracy and precision within historical values is attained the analyst is allowed to work on project samples.

9.0 QUALITY CONTROL SAMPLES

Each project is unique and therefore in each work plan, the numbers and types of blank, duplicate, and spiked samples will be specified. The frequency of these analyses must be related to the purpose of the study, but the following serve as general guidelines for the development of the quality control portion of the work plan.

At the initiation of an analytical procedures for trace organics, the apparatus and the reagents necessary for analysis of samples will be assembled and a series of blank determinations will be performed. These blank determinations will involve all reagent solvents and glassware as required in a standard method of analysis, and will determine when background peaks are sufficiently low (or absent) to permit the analysis of the sample to proceed. If satisfactory blanks are not obtained in these initial samples, then additional steps will be taken to determine the cause and eliminate it. Once the initial methodology has been established, then blank determinations will be performed as a routine procedure throughout the program as samples are analyzed. A blank sample will normally be analyzed along with every ten samples.

The following tests will normally be run with each batch of samples for analysis:

- one method blank
- one standard at a median concentration
- one spiked samples for determination of recovery
- duplicate analyses of one samples from the batch for repeatability

Other sample types and their uses are given in the following table.

Type	Purpose
Field Blank	Evaluate handling and shipping procedures
Field Spike	Evaluate laboratory performance
Laboratory spike into sample	Determine matrix effects on recovery

If appropriate to the purpose of the study, these will be incorporated into the work plan and implemented.

The use of duplicate samples provides assurance that the methodology is performing within previously established limits of accuracy and precision. The frequency of duplicate analyses on samples or repeated injections/aspirations of extracts should be related to the purpose of the program and the eventual end use of the data, but should not be less than ten percent of the samples.

10.0 METHOD VALIDATION

If a method is to be used for which no accuracy and precision statements are available and with which this laboratory has no familiarity, then a series of samples will be run to determine these characteristics. The following program is patterned after "Analytical Quality Control".

The study should include concentrations at three different levels; near the sensitivity limit, near the upper bound of expected values, and inbetween the two. Seven replicate analyses of each concentration should be made, with all the normal procedural steps involved. The recommended order of analysis is seven sequences of low, medium, then high concentrations, which allows for the maximum interferences during operation.

Accuracy and precision statements will be devloped from these data. These will take the form:

> "Recoveries of_____, _____, and _____%, respectively were obtained using this method at concentrations of _____, _____, and _____."
>
> "Standard deviations of _____, _____, and _____, were obtained, respectively, using this method at concentrations of _____, _____, and _____."

For calculation of average percent recovery and associated standard deviations, consult the EPA Quality Control manual.

11.0 QA OBJECTIVES

The overall measurement objective is to characterize waste from the manufacture of organometallic compounds, using criterion established under the Resource Conservation and Recovery Act (RCRA; 40 CFR Part 264, Subpart O). At the present time, the Agency has not established quantitative guidelines as to the precision, accuracy, completeness, representativeness and/or comparability criteria that must be met. However, some specific numerical QA objectives for accuracy and precision of the sample preparation and analysis procedures used in the analytical laboratory have been developed. These objectives are outlined in Table I. The majority of methods to be used have been described in Test Methods for Evaluating Solid Waste, Manual SW-846 and SW-846B, U.S. Environmental Protection Agency, May, 1980, Standard Methods, EPA Chemical Analysis of Water and Wastes and EPA Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater.

These guidelines are based on laboratory experience in applying comparable procedures to a variety of complex sample matrices. If the QA objective given in this section are not achievable because the sample matrices are highly variable and complex, revised objectives will be formulated.

- Accuracy is defined in QAMS-005/80 as the degree of agreement of a measurement or average of measurements with an accepted or true value. Our accuracy goals for this project are to use reference materials of highest, known purity for calibrations and spiking so that determinate errors can be corrected for, and so that the primary uncertainties in the analytical data are due to random errors. The QA objectives are expressed in the following parameters.

  - Reference Materials: All reference materials used as calibration standards or surrogate compounds will be the highest purity commercially available.

  - Instrument Performance: Each instrument used in this project will be checked on each day that samples are analyzed to demonstrate performance. One of the QA objectives for GC/MS is that the average response to the

TABLE 1 - PRECISION GOALS FOR ANALYSIS

Technique or Parameter	Matrix	Method	Precision Rel. Std. Dev.	Completenes
Proximate				
Thermal Gravimetric Analysis	aqueous waste	ASTM[1]	<20%	90%
Loss on Drying, Loss on Ignition	solid, sludge slurry	ASTM[1]	<20%	
		Standards Methods[3]		
Elemental Content	flue gas dust	ASTM[2]	<20%	
Survey				
GRAV		EPA-600/7-78-201	<20%	90%
TCO		EPA-600/7-78-201	<20%	
IR	aqueous waste	EPA-600/7-78-201	Not Applicable	
LRMS	solid, sludge slurry	EPA-600/7-78-201	Factor of 3	
VOC	flue gas dust	EPA-600/7-78-201	<20%	
GC/MS		EPA[5]	<50%	
Directed				
GC/MS - extractables		EPA[5] or EPA[6]	<30%	90%
GC/MS - volatiles	aqueous waste	EPA[5] or EPA[6]	<30%	
HPLC	solid, sludge, slurry	EPA[5] or EPA[6]	<30%	
AAS	flue gas dust	EPA[5] or EPA[6]	<20%	
ICAP		Fed Reg[4]	<20%	
Characteristic				
Corrosivity	aqueous waste	EPA[5]	<20%	90%
Ignitability	solid, sludge, slurry	EPA[5]	<20%	
EP Toxicity	flue gas dust	EPA[5]	<20%	

[1]American Society for Testing and Materials, Annual Book of ASTM Standards, Method D-1888-78 Part 31 (1979)

[2]ASTM, Annual Book of ASTM Standards, Methods: Carbon, Hydrogen D-3178-73 (1979) Part 26; Nitrogen D-3179-73 (1979) Part 26, E-258-67 (1977) Part 30; Oxygen D-3176-74 (1979) Part 26; Sulfur D-3177 (1975) Part 26, D-129-64 (1978) Part 40; Chlorine D-2361-66 (1978) Part 26, D-808-63 (1981) Part 23; Phosphorus D-2795-65 (1980) Part 26

[3]American Public Health Association, American Water Works Association, Water Pollution Control Federatio Standard Methods for the Examination of Water and Wastewater, 15th Edition, 1980, Methods 208A, E

[4]Federal Register, 12/3/79, Part III Envirnmental Protection Agency, Guidelines Establishing Test Procedures for the Analysis of Pollutants; Proposed Regulations. Appendix IV

[5]Test Methods for Evaluating Solid Waste, SW 846 & 846B, May 1980, U.S. EPA.

[6]Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA-600/4-82-057, July 1982

internal standard will not decrease by more than 50% on a daily basis. Otherwise, retuning and/or other corrective action will be implemented.

- Recovery of Surrogates: The recovery of a surrogate compound(s) added to a sample will be defined as follows:

$$\text{Recovery, \%} = \frac{\text{ug S found in sample}}{\text{ug S added to sample}} \times 100 \quad (S = \text{surrogate})$$

This assumes that the surrogate is not present in the sample. The mean and standard deviation of the recovery data will be compiled on a cumulative basis for each surrogate compound in each sample matrix. The objectives for recovery of surrogates are listed in Table 2. Surrogate recovery objectives will be reassessed, if necessary, based upon chemical similarity to the specific analytes.

- Recovery of Metals and Organometallic Compounds: The recovery of metals (M) and/or metal organic compounds will be defined as follows:

$$\text{Recovery, \%} = \frac{\text{(ug M found in spiked sample} - \text{ug M in native sample)}}{\text{ug M added to sample}} \times 100$$

There are no specific numerical targets for recovery of metals and/or organo-metallic compounds from the waste sample, although it is expected that these will approximate the recoveries given above for surrogates. From laboratory experience, it is expected that the recovery of metal species will probably exceed fifty percent (50%).

- Precision is defined in QAMS-005-80 as a measure of mutual agreement among individual measurements of the sample property. The QA objectives for precision are expressed in the following parameters:

- Analysis of Standards: The correlation coefficient for each calibration curve, must be >0.90.

- Analysis of Surrogates: The standard deviation for analysis of surrogate compounds in replicate samples from a given waste stream be within the limits specified in Table 1.

- Analysis of Replicate Samples: The results of survey analysis of laboratory replicate samples must be within the limits specified in Table 1, when at least three replicate samples are analyzed. At least 10% of all analyses performed will be triplicate QC checks.

Recovery Objectives of Surrogates

Sample	Mean	Std Deviation
aqueous liquids	$\geq$ 70%	$\leq$ 30%
organic liquids and solids	$\geq$ 50%	$\leq$ 50%

- Completeness. The objective is to obtain analytical results for at least 90 percent of the samples, based upon the number of samples collected.

- Representativeness. These factors will be addressed in each sampling and analysis plan to insure representative samples: sampling sites, process cycles, batch flow rates (sampling frequency), sample preservation, sampling procedures and equipment.

- Comparability. All data will be reported in mg of analyte per liter of original sample. Recovery information will be provided in conjunction with concentration data.

12.0 DATA REDUCTION AND VALIDATION

- Data Reduction

Data collected from the analysis procedure will consist of peak areas for the surrogate species of analytes of concern. Peak areas will be converted to concentrations with a calibration curve relating the peak area of standards to their concentration. Calibration curves will be constructed by fitting a linear regression equation to the peak areas of the standards at five concentrations.

The raw data will be converted to the concentration of the analyte in the sample by software in our Hewlett Packard data system or by the analyst. From peak areas for the series of known calibration standards a regression line will be computed. Peak areas from the analyses of unknowns will be used to calculate corresponding quantities of analyte from the regression line. The data summary will be included in the analyst's notebook and copies will be forwarded to the Analysis Coordinator.

- Chromatographic and Mass Spectrometry Analyses. An internal standard will be added to each standard solution or concentrated sample extract immediately prior to analysis. The quantity added will be sufficient to give the same concentration (mg/l) of internal standard in all solutions/extracts analyzed. The internal standard will be used to correct for variations in instrument response and sample volume introduced by determining A', the corrected peak area:

$$A' = \frac{\text{uncorrected area of analyte}}{\text{uncorrected area of internal standard}}$$

The factor A' will be used to correct the peak area of each analyte in each standard, surrogate and sample.

Blank corrections will be made by subtracting A' for the method blank from A' for the standard.

Blank-corrected relative areas will be used to calculate the regression equation of the calibration curve.

No correction will be made based upon the recovery of surrogates; surrogate recovery data will be reported along with the results of each set of samples.

- Data Validation

The principal criteria that will be used to validate the data during collection and reporting are:

- Verification on a regular basis during analyses by the QC and Data Manager that all raw data have been referenced in the laboratory chain-of-custody records.

- Examination of at least 5% of the raw data (e.g., chromatograms, AAS recorder outputs) by the Analysis Coordinator to verify adequacy of documentation, and confirm the analyst's data reduction and interpretation methods.

- Confirmation that uncorrected areas for internal standards have not decreased by more than 50% on a daily basis without retuning or implementing other corrective action

- Reporting of all blank, standard, and QC data along with resuts for each batch of samples.

13.0 PERFORMANCE AND SYSTEM AUDITS

Because of the anticipated difficulty in obtaining reference samples with matrices similar to actual waste samples, performance audits will rely heavily on the replicate analyses of real samples, spiked and unspiked. However, we will be alert to opportunities to use standard reference materials as a means of auditing performance.

During the course of system audits, the Quality Assurance Coordinator will remain sensitive to the possible need for additional peer review of any aspect of the program, and he or she will suggest the inclusion of other appropriate York staff in the audit process whenever possible.

14.0 PREVENTIVE MAINTENANCE

Regularly scheduled maintenance of GC, and GC/MS hardware includes replacement of gas purification traps, injector septa and pump seals, along with conditioning each column used. These will be performed according to manufacturer's recommendations or more frequently depending upon the types of solvents used and sample matrices encountered.

Other maintenance, such as cleaning of a fouled injection port or detector will be performed when high blanks, loss of peak resolution, decreased sensitivity or other symptons indicate that a problem exists or QC check criteria are not achieved.

15.0 CORRECTIVE ACTION

For each analytical method employed, precision and accuracy will be regularly tracked by computing the standard deviation of the range of the results of replicate analyses. Periodic determination of recovery of the surrogates will be tabulated. The mean recovery and the standard deviation of replicate sets will be computed. When either the relative standard deviation of replicate results, the average recovery, or the relative standard deviation of replicate results, the average recovery, or the relative standard deviation of replicate recoveries exceeds the performance goals, corrective action will be taken to improve performance prior to analysis of the next lot.

If during system or performance audits, weaknesses or problems are uncovered, corrective action will be initiated immediately.

Corrective action will include, but not necessarily be limited to recalibration of instruments using freshly prepared calibration standards; replacement of lots of solvent or other reagents have unacceptable blank values; additional training of laboratory personnel in correct sample preparation and analyses methods; and reassignment of personnel, if necessary, to improve the overlap between operator skills and method requirements.

Whenever a long-term corrective action is required to eliminate nonconformance, the following closed-loop system* will be used to implement the corrective action procedure and verify its effectiveness:

- The problem will be defined,
- Responsibility for investigation will be assigned,
- The cause of the problem will be investigated and determined,

*Reference: "Quality Assurance Handbook for Air Pollution Measurement Systems. Volume 1: Principles" EPA-600/4-76-005. January, 1976.

- Responsibility for implementing corrective action will be assigned and accepted,
- The action will be implemented and its effectiveness evaluated,
- Verification that the action has eliminated the problem will be established.

SAFETY PLAN
for
CONFIRMATION STUDY ON
HAZARDOUS WASTE SITES
at
NAVAL EDUCATION TRAINING CENTER
NEWPORT, R.I.

November 4, 1983

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Comm. No. 502-10

A/E Contract No. N62472-83-C-1154

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A. INTRODUCTION

1. Background

This Safety Plan covers the safety and health aspects of the confirmation study on hazardous waste sites at the Naval Education and Training Center, (NETC), Newport, Rhode Island under A/E Contract No. N62472-83-C-1154. The overall program of activities to be carried out under the contract is covered in the plan of action. The primary activities will be sampling and laboratory analysis of suspected hazardous wastes in drums; on-shore soil, surface water, ground water and landfill leachate; and off-shore sediment, and mussels. The purpose of the contract is to determine whether or not specific toxic or hazardous materials have contaminated the environment; consequently, hazardous wastes may be encountered during the investigations. As appropriate, this plan covers the necessary training, precautions, procedures and equipment to prevent injury or harm to A/E contractor personnel, Navy personnel and the public during the course of all activities under this contract. Laboratory safety is covered in a separate plan included in the Appendix.

B. POTENTIAL HAZARDS AND PREVENTATIVE MEASURES

1. Types of Hazards

The principal safety hazards under this contract will be those associated with use of sampling equipment and travel by automobile and boat during sample collection and transportation of samples to the laboratory. The samples to be collected include soils; sediments; mussels; liquids such as surface water, ground water and landfill leachate; and suspected hazardous wastes from drums. The principal health hazards include potential for ingestion, inhalation or body contact with gases, fumes or liquids during sampling of suspected hazardous wates in drums; inhalation of dust or contact with the soil during sampling of soil; and possible hazards during sub-surface sampling (soil and groundwater) due to unknown presence of utilities (piping, electrical conduits) or hazardous waste deposits.

2. General Precautions

This Safety Plan is based on the requirements of the Corps of Engineers manual EM385-1-1 "General Safety and Health Requirements Manual" dated April, 1981. The applicable provisions of this manual are a part of this Safety Plan. In addition, the "Notice to Contractor" on accident prevention (6 September 1979) is a part of this Safety Plan (see Appendix).

All motor vehicle regulations will be observed, including those of the State of Rhode Island, those in EM385-1-1 and those governing use of motor vehicles at NETC.

All applicable boating regulations will be observed, including those in EM385-1-1 and those in the United States Coast Guard boating safety regulations.

If the alternative sampling method employing diving is used, diving operations will be carried out in accordance with Em385-1-1 except that:

a. The annual re-qualification requirement of 26.F.02 will not be required of divers.

b. For diving depths up to 33 feet, the diving crew will consist of one diver and one standby diver who will act as Diving Master; a safety line will be employed as provided in 26.F.18.

There are no special safety or health hazards associated with the shipment of any environmental samples; normal precautions will be taken to prevent leakage or damage to sample containers. Samples of hazardous wastes taken from drums will be handled in accordance with federal regulation 40 CFR 261.4(d)(2) (see Appendix). This regulation exempts samples from all hazardous waste regulations provided certain conditions are met.

3. Preventative Measures

a. Sampling of Drums

Although this contract deals with confirmation of the effects of hazardous wastes, only during sampling of drums will raw hazardous wastes be encountered during the planned program. Until the contents of the drums are verified, any activities around the drums will take cognizance that serious safety and health hazards may exist due to:

(1) Fire or explosion of the contents of drums.

(2) Toxicity due to inhalation, ingestion or body contact with contents of the drums.

(3) Spillage of materials in the drums.

The use and manipulation of sampling equipment for drums does not present any special risks which may cause mechanical injury but the hazards listed above must be recognized.

The person doing the sampling will wear coveralls, face shield and rubber gloves. Drums will be opened using non-sparking tools and the

Coliwasa sampler will be constructed of non-sparking materials. The transfer of the samples into the sampling containers will be conducted in or over a containment vessel (bucket or tub) to prevent spillage onto the ground surface. Appropriate personal hygiene precautions will be taken during sampling to avoid personal contact with the waste and to prevent ingestion or inhalation of the waste or any fumes, dust or gases emanating from it.

b. Sampling of Soils

The soils to be sampled may be contaminated with hazardous waste constituents but the concentrations of these constituents is expected to be low and health and safety risks are low. The use and manipulation of soil sampling equipment does not present any special risks which may cause mechanical injury. However, if dusty conditions are evident, respiratory and eye protection will be worn. A Dig-Safe permit will be obtained before making any excavations.

Since soil samples will be collected in areas where it is suspected that hazardous wastes may have been buried, it is possible that such wastes may be encountered during soil sampling. This is considered to be unlikely; however, sampling activities will be discontinued if there is any evidence that any material other than soil is encountered. Sampling will not be resumed until the nature of the material is evaluated and it is determined that the sampling can proceed safely.

c. Sampling of Sediments and Mussels

These samples may contain hazardous waste constituents but, if present, the concentrations will be too low to constitute a handling hazard. The use and manipulation of the sampling equipment does not present any special risks which may cause mechanical injury. However,

precautions will be necessary to prevent boating accidents during manipulation of sampling equipment over the side of the boat. If the alternative sampling method employing diving is used, diving operations will be carried out as described above.

d. Sampling of Ground Water

Collection of samples of ground water will require subsurface drilling and setting of monitoring wells. Drilling operations present the risk of striking underground utilities, piping or electrical conduits. The only drilling planned in the first phase of work will be near abandoned buried oil storage tanks at Tank Farm 4. These tanks were emptied and filled with water several years ago. The tanks were not used for storage of low flash point oils so that very low hazard exists for fire or explosion during drilling operations due to the presence of flammable materials which may have leaked into the ground or which may be present in unknown abandoned pipe lines.

Sub-contractors engaged to perform the required drilling and setting of monitoring wells will be required to comply with this safety plan and will be provided with necessary background data on the site. All known data on underground piping and utilities will be obtained and plotted on large scale drawings to form the basis for selection of drilling locations and filing for a Dig-Safe permit.

The use and manipulation of the sampling equipment does not present any special risks which may cause mechanical injury.

e. Sampling of Surface Water and Leachate

These samples may contain hazardous waste constituents but, if present, the concentrations will be too low to constitute a handling hazard. The use and manipulation of the sampling equipment does not present any special risks which may cause mechanical injury.

C. DESCRIPTION OF ACTIVITIES OF EFD AND FACILITY PERSONNEL WHICH MAY INCLUDE EXPOSURE TO POTENTIAL HAZARDS

Navy personnel will not participate in the actual sampling activities although they may be present during the sample collection program. Consequently, they may be exposed to any of the hazards discussed in Section B including hazards due to use of motor vehicles, boating, sample collection, and monitoring well installation.

D. COORDINATION AND TRAINING OF EPD AND FACILITY PERSONNEL AND A/E PERSONNEL

All activities at NETC will be coordinated through Mr. Thomas Sheckels of NORDIV and Mr. Martin Dwyer of NETC. These are the only Navy personnel who may be exposed to any hazards in connection with this program. This Safety Plan will be reviewed with them prior to commencement of any sampling or other related operations. Prior to the start of any sampling, all personnel, including Navy personnel, will be briefed concerning specific potential hazards and precautions required at each site.

E. PERMITS

The only permit specifically required for safety and health purposes is a Dig-Safe permit. A copy of the plan of action will be submitted for the purpose of obtaining the Dig-Safe permit. In addition, a large scale drawing will be submitted for specific sites where excavations or drilling are planned.

All activities will be scheduled in advance with notifications to NORDIV (Mr. Thomas Sheckels) and NETC (Mr. Martin Dwyer) so that any advance internal notifications needed for security or safety reasons can be made.

F. EMERGENCY RESPONSE PROCEDURES AND NOTIFICATIONS

1. Emergency Response Procedures

The initial response to any emergency will be to protect human health and safety, and then the environment. If there is a continuing danger to personnel, the immediate area will be evacuated. First aid will be given to any injured personnel; a first aid kit will be available at all times while sampling activities are in progress. If a spill of hazardous waste has occurred, efforts will be made to contain it, if this can be done without endangering human health or safety. If a fire or explosion is involved, the appropriate fire department will be notified; no unusual efforts will be made by A/E personel to fight fires.

2. Notifications

Notifications will be made as soon as possible for all emergencies requiring fire, police or ambulance service. The NETC telephone numbers for these services are as follows:

Fire	841-3333
Police	841-3241
Ambulance	841-2222

All accidents involving personal injury or property damage exceeding $300 in value will be reported as follows:

a. Telephone report as soon as possible to Mr. Martin Dwyer of NETC Public Works Dept.,

Telephone: 401-841-2061

b. Written report on OSHA Form No. 101 (see Appendix) within 6 days to:

Mr. Martin Dwyer

Address: Public Works Department
Naval Education and Training Center
Newport, Rhode Island 02841
Attention: Code 42P

and to Mr. Thomas Sheckels, NORDIV

Address: Commanding Officer
Northern Division
Naval Facilities Engineering Command
Building 77L
Philadelphia Naval Base
Philadelphia, PA 19112
Attention: Code 114.

c. A record will be kept of all incidents requiring first aid; a copy of this record will be submitted to NETC or NORDIV upon request. This record will be kept on the First Aid Record form (see Appendix).

APPENDIX A

ANALYTICAL LABORATORY SAFETY PROGRAM

YORK WASTEWATER CONSULTANTS, INC.

ANALYTICAL LABORATORY SAFETY PROGRAM

LABORATORY SAFETY

The arrangements for laboratory safety in a given facility may be chosen from one of several alternatives as appropriate to the physical size, numbers of staff and nature and range of hazards encountered.

At York, laboratory safety is under the direction of the Laboratory Safety Officer. This individual is knowledgeable and experienced in all the day-to-day operations of the laboratory and possesses fundamental management characteristics necessary for strict enforcement of safety rules.

Our laboratory safety officer is the Chief Chemist whose duties in addition to technical direction include keeping a watchful eye on the day-to-day activities in the laboratory and keeping abreast of developments in the safety field and develop new safety arrangements as required.

General Attitude to Laboratory Safety

The attitude of staff members toward laboratory safety is directly related to its success. Most accidents are related to housekeeping problems. An untidy laboratory is a direct encouragement to staff to become slack in their general attitude to safety. To eliminate this problem, 15-20 minutes at the end of each day are set aside to clear work spaces of unnecessary glassware and equipment. In addition, once per week, one full hour is spent cleaning work benches of other unnecessary items.

Fire Prevention

The annual cost of fire losses in laboratories has been and is steadily rising (Cooke, A.J.D. 1976, A Guide to Laboratory Law, 1976). Many fires arise from simple oversights such as instrumentation or other electronic equipment left on overnight, self-ignition of packing materials, etc. At York we have a code of practice for the prevention of fire whose secondary aim is to increase the chance of successful containment of fire once it has started so that the resultant damage may be reduced.

The Safety Officer at York minimizes fire chances through enforcement of the following key items:

- Daily removal of rubbish;
- Storage and disposal of combustible packing material to a safe (isolated from lab operations) place;
- Storage of solvents (flammable) in explosion proof cabinets;
- Relay information to staff on fire hazards of new materials from storage and use viewpoints;
- Proper disposal of waste solvents;
- Monitoring of electrical connections and wiring;
- Smoking is confined to appropriate areas;
- Non-laboratory personnel traffic is kept to a minimum;
- Routine checkout of fire/smoke detectors and alarms;
- Routine checkout of fire-fighting equipment including foam, CO_2, powder and CCl_4 extinguishers;
- Semi-annual fire-drill initiation.

Cylinders of Compressed Gas

The number of gas cylinders in use in our aboratory is kept to a practicable minimum. They are always firmly fixed or supported by means of chains or straps and are never used in situations where the temperature is likely to rise significantly, e.g. near radiators, in direct sunlight or in hot-rooms.

Cylinders of compressed gas are also always used with the appropriate control heads or pressure regulators together with suitable non-return valves and, where apprpriate, flame traps. Control heads, pressure regulators and non-return valves are

never oiled or greased. If connected to any thin-walled metal appratus (and certainly when connected to any apparatus constructed of glass or plastic) there is provision for automatic pressure release so that the apparatus is not submitted to undue stress.

Cylinders are always transported in properly constructed cylinder trolleys and never dropped.

Cryogenic Substances

Our laboratories use liquid gases or cryogenic mixtures such as solid carbon dioxide, and liquid nitrogen, in order to produce low temperatures in cooling baths or traps. These liquids and mixtures may produce very painful and severe burns and destruction of tissue if allowed to come into contact with the body, and therefore suitable protective clothing is worn when handling them. Gloves, face masks and aprons are provided. Rubber boots, if provided, are worn inside trousers to prevent spilled liquid falling into the boot which may be extremely difficult to remove quickly in the event of a spillage. For the same reasons it is inadvisable to wear gauntlet-type gloves.

There are a number of precautions that are observed when using cryogenic substances.

(1) Liquid nitrogen, solid carbon dioxide (or mixtures containing it) should not be used or stored in confined spaces or rooms with little or no ventilation, since there is considerable risk that the air in such a room will be rapidly depleted of oxygen by the release of large volumes relatively inert gas. It is not unknown for laboratory workers to store blocks os solid carbon dioxide in cold-rooms, which generally have very little ventilation. This practice will very soon produce a suffocating atmosphere in the room.

(2) Liquefied gases and other very cold liquids must not be poured into unsuitable containers. The thermal shock produced may well be sufficient to shatter the container; many of the vacuum flasks produced for domestic purposes,

for example, are incapable of withstanding the very sudden lowering of their temperature when liquid nitrogen is poured into them.

(3) Liquefied gas must not be stored in containers or vessels that are not freely vented to the atmosphere. There is a danger that gas released by evaporation from the liquid will be unable to escape from the vessel at a sufficient rate to prevent the vessel becoming pressurized and subsequently exploding. This is particularly important with narrow-necked, metal, vacuum-insulated storage containers. In humid conditions, it is possible that ice may form at the mouth of the container and this would severely restrict the outflow of escaping gas.

Chemicals

There are many hazards associated with the use and handling of chemicals.

Chemicals are used by workers in many fields, some of whom may have had little or no training in chemistry. Many substances in common use are highly toxic and their use by inexperienced persons is carefully supervised.

Occupational Hygiene

In all laboratories in which hazardous (toxic or infectious) substances are used, a high standard of cleanliness and personal hygiene must be maintained.

Eating, drinking, smoking, the application of cosmetics, licking of labels, mouth pipetting, chewing of pencils, biting of fingernails, or any activity which could lead to the ingestion of toxic substances, is prohibited. Since chemicals may also enter the body by inhalation and by absorption through the skin and eyes, particular care is exercised when weighing or transferring toxic substances in dry powder form from one container to another.

The Laboratories in which toxic substances are handled are well ventilated and provided with sufficient properly designed

properly designed and constructed fume hoods, suitable washing (eye, shower) facilities with hot and cold water, and an adequate supply of disposable paper towels.

For staff working with highly corrosive chemicals, it is necessary to wear goggles or face masks and rubber or plastic gloves. If the work being carried out involves the use of more than small amounts of corrosive substances, it is necessary to wear rubber or plastic aprons and boots. All staff is trained so that full use is made of the facilities provided for any such emergencies. The Safety Officer ensures that all spillages are effectively cleared up immediately after they occur and that a suitable standard of cleanliness is maintained at all times. Laboratory coats, are worn by all laboratory staff while working and they are removed before entering other rooms where food is consumed.

Dermatitis and Skin Reactions

Many substances, such as chromium salts, chlorinated hydrocarbons, phenolic compounds and animal hair and dust, will produce skin reactions and lesions which are not only of unpleasant appearance, but are often extremely painful and slow to heal. Sensitivity to these substances varies enormously between individuals. Many persons are liable to become so sensitized that after receiving a few fairly short initial exposures to the substance their response to a given dose or exposure becomes much greater and the result more painful. If they are isolated from the substance they will generally recover, but a subsequent brief exposure may well produce an immediate and severe response. At this stage, they may have to be removed permanently from the work that brings them into contact with the substance to which they have become sensitized.

Chromic acid which is widely used for cleaning laboratory glassware is still, regrettably, a common cause of contact dermatitis. Prolonged exposure of the hands and forearms to chromates will produce deep, sharply defined ulcers that are slow to heal. In our laboratories, chromic acid has now been replaced by modern powerful detergents specially formulated for cleaning glassware and their use has done much towards reducing the incidence of contact dermatitis. Because of the nature of

their work, laboratory glassware cleaning personnel are especially protected from the effects of these harmful substances. If they are allowed to put their bare hands into hot water containing detergent, their skin may become defatted and thus be more easily penetrated by the causative agents. The Safety Officer ensures that the glassware cleaners wear rubber or plastic gloves when they are working and that they report any sign of skin irritation immediately.

Toxic Substances and Threshold Limit Values

Some countries, notably the UK and the USA, have introduced legislation or regulations (OSHA) that define the maximum concentration of a substance in air to which a person may be exposed whilst at work. Threshold limit values (TLVs) refer to a time-weighted concentration for a 7 or 8-hour working day or a 40-hour working week. TLVs are expressed as parts per million (ppm) or milligrams per cubic metre (mg/m^3). Time-weighted averages generally permit excursions for a limited period above the limit, provided that it is compensated for by excursions below the limit. Some substances have ceiling limits above which no excursion is permitted. The tables for TLVs are based on the best available information from industrial experience and from experimental human and animal studies.

The sense of smell is a useful, but not a very reliable, guide to the concentration of the substance present. It should be noted that it is possible for two substances of modest toxicity to combine chemically or react to produce an extremely toxic product, e.g. the TLV of chlorine is 0.1 and that of formaldehyde is 2.0. It is possible, but not very likely, that under certain conditions these two substances could react together to form the extremely carcinogenic substances bis-chloro-methyl-ether, which has a TLV of 0.001 ppm. Therefore, all OSHA noted substances are used with proper ventilation considerations.

Carcinogens

A number of substances have been associated with an increased risk of the development of neoplastic disease in both man and animals. More are suspected of carcinogenic action and many more remain as yet untested for this type of activity. A

number of substances whose use has been suggesed as a safe substitute for known or suspected carcinogens have later been found to be almost as active in this respect as the original material.

With new substances being added almost daily to the list of compounds known to have, or suspected of having carcinogenic properties, it is imperative that all laboratory staff should be aware of the hazards and that adequate precautions are taken whenever these substances are used or handled in the laboratory.

It has been suggested by Howe (Laboratory Practice, 24, 457-567, 1975) that, as far as animal experiments are concerned, it is possible to grade the available evidence about carcinogens into three arbitrary categores:

(1) Potent carcinogens - strong, proven carcinogens associated with high incidence of cancer.

(2) Carcinogenic - proven carcinogens with moderate or weak activity.

(3) Suspect carcinogens - inconclusive evidence of carcinogenicity or untested compounds structurally related to known carcinogens.

Compounds falling within the first group require precautions at facilities that are unlikely to be available or possible in many laboratories. The manufacturer, importation and sometimes the use of these substances has been restricted or prohibited in many countries.

In general, the risk of developing neoplastic disease from working with carcinogenic substances is in proportion to (a) the length and frequency of exposure, and (b) the amount or concentration of the substance to which one is exposed.

Irrespective of the type and quantity of equipment that is provided for the safe handling of carcinogenic substances, and of the rigour with which the precautionary measure are enforced, a very high standard of occupational hygiene is maintained when and wherever they are used or handled.

Many codes of practice have been published for the safe use of carcinogens. We advise staff to avoid the use of these substances altogether and, if this is not possible or reasonably practicable, to substitute safer and less hazardous compounds whenever possible.

It is the duty of the Safety Officer to ensure that all dangerous substances arising from work in the laboratories are disposed of safely, and with due regard to any local or other legal requirement, such as manifests (RCRA), etc.

OFFICER-IN-CHARGE OF CONSTRUCTIO
NAVAL ACTIVITIES
NARRAGANSETT BAY AREA
NEWPORT, RHODE ISLAND 02840

NOTICE TO CONTRACTOR 6 SEPTEMBER 1979

Subject: ACCIDENT PREVENTION

1. General Provisions 45 entitled ACCIDENT PREVENTION (JUN 77) is applicable to your recently awarded contract. Accordingly, a SAFETY PLAN must be submitted by your firm before the start of construction.

2. Your responsibilities in this area are outlined in the attached "SAFETY PROGRAM".

3. When reporting an accident, you are required to submit three (3) copies of completed OSHA FORM NO. 101 or other acceptable forms to the Project Engineer furnishing all necessary data - as detailed in the attached Program. One copy of the 101 form is attached for your convenience and may be reproduced.

4. Procedures for compliance will be reviewed with you during the Pre-Construction Conference.

NOTICE TO CONTRACTOR

OFFICER-IN-CHARGE OF CONSTRUCTION
NAVAL ACTIVITIES
NARRAGANSETT BAY AREA
NEWPORT, RHODE ISLAND 02840

In reply refer to:

SAFETY PROGRAM

The Naval Facilities Engineering Command has established an intensive safety program in an effort to provide for and assure, insofar as humanly possible, safe and healthful working conditions for each and every person engaged in NAVFAC contract construction operations. This provides for savings to the Government, savings to your company, and the preservation of our human resources. This letter outlines your responsibilities in connection therewith. Contract safety compliance shall be in accordance with the applicable provisions of your contract which include the Corps of Engineers Safety Manual (EM 385-1-1) of ~~1 June 1977~~ APR 1981 entitled "General Safety Requirements".

SAFETY PLAN

If applicable to the subject contract, clause 45 of the General Provisions entitled Accident Prevention requires that a Safety Plan be submitted before start of construction. The following items should be included in the plan for approval by the Officer in Charge of Construction/Resident Officer in Charge of Construction.

a. Name of the safety representative. State his qualifications and delineate his authority to direct work stoppage and expend funds to eliminate imminent danger conditions.

b. State the frequency at which recorded safety inspections will be conducted by the safety representative and provide a sample of the safety inspection checklist report.

c. State company plan for initial safety indoctrination of all employees.

d. State company plans for continued safety education for all employees including weekly safety meetings.

OFFICER-IN-CHARGE OF CONSTRUCTION
NAVAL ACTIVITIES
NARRAGANSETT BAY AREA
NEWPORT, RHODE ISLAND 02840

In reply refer to:

e. State plans for the posting of proper procedures for the reporting of fire or medical emergencies as well as the telephone numbers of these services.

f. State company housekeeping plans.

g. Provide a statement of hazards expected to be encountered and the proposed method of guarding or correction.

h. Provide a statement that the applicable provisions of the Corps of Engineers Safety Manual will be complied with.

REPORT OR ACCIDENT

You are required to prepare a record, within six (6) days, of each recordable occupational injury, illness and/or property or equipment damage in excess of three hundred dollars ($300). OSHA Form No. 101, Workers' Compensation forms, insurance forms, other forms or supplements, or even a seperate plain sheet of paper may be used to report each accident as long as such record contains all the specific facts precribed below.

a. The name and address of employer

b. The name, home address, age, sex and occupation of employee

c. The activity and exact location of accident

d. Describe the injury or illness in detail and indicate part of the body affected (e.g., amputation of right index finger at second joint)

e. Probable disability - Death, permanent total, permanent partial, temporary total, or temporary partial

f. Describe in detail how the accident occurred

g. Describe what has been done to prevent recurrence

h. Date of injury, illness, and/or property damage and date injured stopped work, if different

i. Date injured returned to work

j. Describe property or equipment damage

k. State who owns property or equipment damaged

l. Estimate dollar value of property or equipment damaged.

OFFICER-IN-CHARGE OF CONSTRUCTION
NAVAL ACTIVITIES
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NEWPORT, RHODE ISLAND 02840

In reply refer to:

You may attach a separate sheet of paper to any of the forms mentioned in the above paragraph in order to provide all the above information, if the form used does not do so.

It is the intent of this Command to aid and assist in every way possible to better ensure an accident-free completed project.

OSHA No. 101
Case or File No. .....

Form approved
OMB No. 44R 1453

Supplementary Record of Occupational Injuries and Illnesses

EMPLOYER

1. Name _____
2. Mail address _____
(No. and street) (City or town) (State)
3. Location, if different from mail address _____

INJURED OR ILL EMPLOYEE

4. Name _____ Social Security No. _____
(First name) (Middle name) (Last name)
5. Home address _____
(No. and street) (City or town) (State)
6. Age _____ 7. Sex: Male_____ Female_____ (Check one)
8. Occupation _____
(Enter regular job title, *not* the specific activity he was performing at time of injury.)
9. Department _____
(Enter name of department or division in which the injured person is regularly employed, even though he may have been temporarily working in another department at the time of injury.)

THE ACCIDENT OR EXPOSURE TO OCCUPATIONAL ILLNESS

10. Place of accident or exposure _____
(No. and street) (City or town) (State)
If accident or exposure occurred on employer's premises, give address of plant or establishment in which it occurred. Do not indicate department or division within the plant or establishment. If accident occurred outside employer's premises at an identifiable address, give that address. If it occurred on a public highway or at any other place which cannot be identified by number and street, please provide place references locating the place of injury as accurately as possible.
11. Was place of accident or exposure on employer's premises? _____ (Yes or No)
12. What was the employee doing when injured? _____
(Be specific. If he was using tools or equipment or handling material, name them and tell what he was doing with them.)
13. How did the accident occur? _____
(Describe fully the events which resulted in the injury or occupational illness. Tell what happened and how it happened. Name any objects or substances involved and tell how they were involved. Give full details on all factors which led or contributed to the accident. Use separate sheet for additional space.)

OCCUPATIONAL INJURY OR OCCUPATIONAL ILLNESS

14. Describe the injury or illness in detail and indicate the part of body affected. _____
(e.g.: amputation of right index finger at second joint; fracture of ribs; lead poisoning; dermatitis of left hand, etc.)
15. Name the object or substance which directly injured the employee. (For example, the machine or thing he struck against or which struck him; the vapor or poison he inhaled or swallowed; the chemical or radiation which irritated his skin; or in cases of strains, hernias, etc., the thing he was lifting, pulling, etc.)
16. Date of injury or initial diagnosis of occupational illness _____
(Date)
17. Did employee die? _____ (Yes or No)

OTHER

18. Name and address of physician _____
19. If hospitalized, name and address of hospital _____

Date of report _____ Prepared by _____
Official position _____

(2) In the case of a waste which is a listed waste under Subpart D, contains a waste listed under Subpart D or is derived from a waste listed in Subpart D, it also has been excluded from paragraph (c) under §§ 260.20 and 260.22 of this Chapter.

§ 261.4 Exclusions.

(a) *Materials which are not solid wastes.* The following materials are not solid wastes for the purpose of this Part:

(1) (i) Domestic sewage; and

(ii) Any mixture of domestic sewage and other wastes that passes through a sewer system to a publicly-owned treatment works for treatment. "Domestic sewage" means untreated sanitary wastes that pass through a sewer system.

[Interim final]

(2) Industrial wastewater discharges that are point source discharges subject to regulation under Section 402 of the Clean Water Act, as amended.

[Comment. This exclusion applies only to the actual point source discharge. It does not exclude industrial wastewaters while they are being collected, stored or treated before discharge, nor does it exclude sludges that are generated by industrial wastewater treatment.]

(3) Irrigation return flows.

(4) Source, special nuclear or by-product material as defined by the Atomic Energy Act of 1954, as amended, 42 U.S.C. 2011 *et seq.*

(5) Materials subjected to in-situ mining techniques which are not removed from the ground as part of the extraction process.

(b) *Solid wastes which are not hazardous wastes.* The following solid wastes are not hazardous wastes:

(1) Household waste, including household waste that has been collected, transported, stored, treated, disposed, recovered (e.g., refuse-derived fuel) or reused. "Household waste" means any waste material (including garbage, trash and sanitary wastes in septic tanks) derived from households (including single and multiple residences, hotels and motels.)

(2) Solid wastes generated by any of the following and which are returned to the soils as fertilizers:

(i) The growing and harvesting of agricultural crops.

(ii) The raising of animals, including animal manures.

(3) Mining overburden returned to the mine site.

(4) Fly ash waste, bottom ash waste, slag waste, and flue gas emission control waste generated primarily from the combustion of coal or other fossil fuels.

(5) Drilling fluids, produced waters, and other wastes associated with the exploration, development, or production of crude oil, natural gas or geothermal energy.

(6)(i) Wastes which fail the test for the characteristic of EP toxicity because chromium is present or are listed in Subpart D due to the presence of chromium, which do not fail the test for the characteristic of EP toxicity for any other constituent or are not listed due to the presence of any other constituent, and which do not fail the text for any other characteristic, if it is shown by a waste generator or by waste generators that:

(A) The chromium in the waste is exclusively (or nearly exclusively) trivalent chromium; and

(B) The waste is generated from an industrial process which uses trivalent chromium exlcusively (or nearly exclusively) and the process does not generate hexavalent chromium; and

(C) The waste is typically and frequently managed in non-oxidizing environments.

(ii) Specific wastes which meet the standard in paragraphs (b)(6)(i)(A), (B) and (C) (so long as they do not fail the test for the charactristic of EP toxicity, and do not fail the test for any other characteristic) are:

(A) Chrome (blue) trimmings generated by the following subcategories of the leather tanning and finishing industry: hair pulp/chrome tan/retan/wet finish; hair save/chrome tan/retan/wet finish; retan/wet finish; no beamhouse; through-the-blue; and shearling.

(B) Chrome (blue) shavings generated by the following subcategories of the leather tanning and finishing industry: hair pulp/chrome tan/retan/wet finish; hair save/chrome tan/retan/wet finish; retan/wet finish; no beamhouse; through-the-blue; and shearling.

(C) Buffing dust generated by the following subcategories of the leather tanning and finishing industry: hair pulp/chrome tan/retan/wet finish; hair save/chrome tan/retan/wet finish; retan/wet finish; no beamhouse; through-the-blue.

(D) Sewer screenings generated by the following subcategories of the leather tanning and finishing industry: hair pulp/crome tan/retan/wet finish; hair save/chrome tan/retan/wet finish; retan/wet finish; no beamhouse; through-the-blue; and shearling.

(E) Wastewater treatment sludges generated by the following subcategories of the leather tanning and finishing industry: hair pulp/chrome tan/retan/wet finish; hair save/chrome tan/retan/wet finish; retan/wet finish; no beamhouse; through-the-blue; and shearling.

(F) Wastewater treatment sludes generated by the following subcategories of the leather tanning and finishing industry: hair pulp/chrome tan/retan/wet finish; hair save/chrome-tan/retan/wet finish; and through-the-blue.

(G) Waste scrap leather from the leather tanning industry, the shoe manufacturing industry, and other leather product manufacturing industries.

(H) Wastewater treatment sludges from the production of TiO_2 pigment using chromium-bearing ores by the chloride process.

(7) Solid waste from the extraction, beneficiation and processing of ores and minerals (including coal), including phosphate rock and overburden from the mining of uranium ore.

(8) Cement kiln dust waste.

(9) Solid waste which consists of discarded wood or wood products which fails the test for the characteristic of EP toxicity and which is not a hazardous waste for any other reason if the waste is generated by persons who utilize the arsenical-treated wood and wood products for these materials' intended end use.

(c) Hazardous wastes which are exempted from certain regulations. A hazardous waste which is generated in a product or raw material storage tank, a product or raw material transport vehicle or vessel, a product or raw material pipeline, or in a manufacturing process unit or an associated non-waste-treatment-manufacturing unit, is not subject to regulation under Parts 262 through 265, 270, 271 and 124 of this chapter or to the notification requirements of Section 3010 of RCRA until it exits the unit in which it was generated, unless the unit is a surface impoundment, or unless the hazardous waste remains in the unit more than 90 days after the unit ceases to be operated for manufacturing, or for storage or transportation of product or raw materials.

[261.4(c) amended by 48 FR 14153, April 1, 1983]

(d) *Samples.* (1) Except as provided in paragraph (d)(2) of this section, a sample of solid waste or a sample of water, soil, or air, which is collected for

the sole purpose of testing to determine its characteristics or composition, is not subject to any requirements of this part or Parts 262 through 267 or Part 270 or Part 124 of this chapter or to the notification requirements of Section 3010 of RCRA, when:

[261.4(d) introductory paragraph amended by 48 FR 30115, June 30, 1983]

(i) The sample is being transported to a laboratory for the purpose of testing; or

(ii) The sample is being transported back to the sample collector after testing; or

(iii) The sample is being stored by the sample collector before transport to a laboratory for testing; or

(iv) The sample is being stored in a laboratory before testing; or

(v) The sample is being stored in a laboratory after testing but before it is returned to the sample collector; or

(vi) The sample is being stored temporarily in the laboratory after testing for a specific purpose (for example, until conclusion of a court case or enforcement action where further testing of the sample may be necessary).

(2) In order to qualify for the exemption in paragraph (d)(1) (i) and (ii) of this section, a sample collector shipping samples to a laboratory and a laboratory returning samples to a sample collector must:

(i) Comply with U.S. Department of Transportation (DOT), U.S. Postal Service (USPS), or any other applicable shipping requirements; or

(ii) Comply with the following requirements if the sample collector determines that DOT, USPS, or other shipping requirements do not apply to the shipment of the sample:

(A) Assure that the following information accompanies the sample:

(*1*) The sample collector's name, mailing address, and telephone number;

(*2*) The laboratory's name, mailing address, and telephone number;

(*3*) The quantity of the sample;

(*4*) The date of shipment; and

(*5*) A description of the sample.

(B) Package the sample so that it does not leak, spill, or vaporize from its packaging.

(3) This exemption does not apply if the laboratory determines that the waste is hazardous but the laboratory is no longer meeting any of the conditions stated in paragraph (d)(1) of this section.

§261.5 Special requirements for hazardous waste generated by small quantity generators.

(a) A generator is a small quantity generator in a calendar month if he generates less than 1000 kilograms of hazardous waste in that month.

(b) Except for those wastes identified in paragraphs (e) and (f) of this section, a small quantity generator's hazardous wastes are not subject to regulation under Parts 262 through 265 and Parts 270 and 124 of this chapter, and the notification requirements of Section 3010 of RCRA, provided the generator complies with the requirements of paragraph (g) of this section.

[261.5(b) amended by 48 FR 14153, April 1, 1983]

(c) Hazardous waste that is beneficially used or re-used or legitimately recycled or reclaimed and that is excluded from regulation by § 261.6(a) is not included in the quantity determinations of this section, and is not subject to any requirements of this section. Hazardous waste that is subject to the special requirements of § 261.6(b) is included in the quantity determinations of this section and is subject to the requirements of this section.

(d) In determining the quantity of hazardous waste he generates, a generator need not include:

(1) His hazardous waste when it is removed from on-site storage; or

(2) Hazardous waste produced by on-site treatment of his hazardous waste.

(e) If a small quantity generator generates acutely hazardous waste in a calendar month in quantities greater than set forth below, all quantities of that acutely hazardous waste are subject to regulation under Parts 262 through 265 and Parts 270 and 124 of this chapter, and the notification requirements of Section 3010 of RCRA:

[261.5(e) amended by 48 FR 14153, April 1, 1983]

(1) A total of one kilogram of commercial chemical products and manufacturing chemical intermediates having the generic names listed in § 261.33(e), and off-specification commercial chemical products and manufacturing chemical intermediates which, if they met specifications, would have the generic names listed in § 261.33(e).

(2) A total of 100 kilograms of any residue or contaminated soil, water or other debris resulting from the cleanup of a spill, into or on any land or water, of any commercial chemical products or manufacturing chemical intermediates having the generic names listed in § 261.33(e), or any residue or contaminated soil, water or other debris resulting from the cleanup of a spill, into or on any land or water, of any off-specification commercial chemical products or manufacturing chemical intermediates which, if they met specifications, would have the generic names listed in § 261.33(e).

(f) A small quantity generator may accumulate hazardous waste on-site. If he accumulates at any time more than a total of 1000 kilograms of his hazardous waste, or his acutely hazardous wastes in quantities greater than set forth in paragraphs (e)(1) or (e)(2) of this section, all of those accumulated wastes for which the accumulation limit was exceeded are subject to regulation under Parts 262 through 265 and Parts 270 and 124 of this chapter, and the notification requirements of Section 3010 of RCRA. The time period of § 262.34 for accumulation of wastes on-site begins for a small quantity generator when the accumulated wastes exceed the applicable exclusion level.

[261.5(f) amended by 48 FR 14153, April 1, 1983]

(g) In order for hazardous waste generated by a small quantity generator to be excluded from full regulation under this section, the generator must:

(1) Comply with § 262.11 of this chapter;

(2) If he stores his hazardous waste on-site, store it in compliance with the requirements of paragraph (f) of this section; and

[261.5(g)(3)(i) — (iii) amended by 48 FR 14153, April 1, 1983]

(3) Either treat or dispose of his hazardous waste in an on-site facility, or ensure delivery to an off-site storage, treatment or disposal facility, either of which is:

(i) Permitted under Part 270 of this chapter;

(ii) In interim status under Parts 270 and 265 of this chapter;

(iii) Authorized to manage hazardous waste by a State with a hazardous waste management program approved under Part 271 of this chapter;

(iv) Permitted, licensed or registered by a State to manage municipal or industrial solid waste; or

(v) A facility which:

(A) Beneficially uses or re-uses, or legitimately recycles or reclaims his waste; or

(B) Treats his waste prior to beneficial use or re-use, or legitimate recycling or reclamation.

(h) Hazardous waste subject to the reduced requirements of this section may be mixed with non-hazardous waste and remain subject to these reduced requirements even though the

RECORD OF FIRST AID

CONFIRMATION STDUDY ON
HAZARDOUS WASTES SITES
at
NAVAL EDUCATION TRAINING CENTER
NEWPORT, R.I.

Loureiro Engineering Associates/York Wastewater Consultants
Comm. No. 502-10
A/E Contract No. N62472-83-C-1154

DATE OF INCIDENT: TIME OF DAY:

NAME AND ADDRESS OF INJURED PERSON:

SOCIAL SECURITY NUMBER:

DESCRIPTION & LOCATION OF INCIDENT:

NATURE OF INJURY OR ILLNESS:

TREATMENT GIVEN:

TREATED BY:

SUPERVISOR: